DOCUMENT RESUME

ED 215 868 SE 037 057

AUTHOR Barron, Marcelline A.
TITLE Chemistry: Coping with Change...Creatively.
PUB DATE 73
NOTE 180p.

EDRS PRICE MF01/PC08 Plus Postage.
DESCRIPTORS *Change; *Chemistry; *Laboratory Manuals; Mathematics Anxiety; Science Activities; *Science Curriculum; Science Education; *Science Experiments; Secondary Education; *Secondary School Science

ABSTRACT

Developed for mathematics-shy high school chemistry students, this laboratory manual is suitable for use with any chemistry textbook. Seventy-three experiments, based on a theme of change, are grouped into 5 general areas: (1) 9 experiments focusing on skills needed in observing reality; (2) 19 experiments interpreting how reality changes, including properties of matter, physical and chemical changes, reaction rates, and catalysts; (3) 11 experiments indicating how models are used to categorize and quantify change; (4) 24 ecochemistry experiments designed to form a model of a changing universe, including such activities as testing water samples, identification of metallic ions, and analysis of materials found in plant tissues; and (5) 10 nutrition/organic/biochemistry experiments to illustrate that change also affects students' collective everyday lives, including consumer food tests for butter, margarine, caffein, synthetic dyes, and citric acid in fruit juices, and the preparation of common organic compounds. (JN)

****
* Reproductions supplied by EDRS are the best that can be made *
* from the original document. *
****

ED215868

U.S. DEPARTMENT OF EDUCATION
NATIONAL INSTITUTE OF EDUCATION
EDUCATIONAL RESOURCES INFORMATION CENTER (ERIC)

✓ This document has been reproduced as received from the person or organization originating it.
Minor changes have been made to improve reproduction quality.

• Points of view or opinions stated in this document do not necessarily represent official NIE position or policy.

"PERMISSION TO REPRODUCE THIS MATERIAL HAS BEEN GRANTED BY
MARCELLINE A. BARRON

TO THE EDUCATIONAL RESOURCES INFORMATION CENTER (ERIC)."

SE 037 057

PERIODIC TABLE OF ELEMENTS

IA	IIA	IIIB	IVB	VB	VIB	VIIB	VIII			IB	IIB	IIIA	IVA	VA	VIA	VIIA	INERT GASES
1 * H																	2 * He 4.00
3 ○ Li 6.94	4 ○ Be 9.01											5 ○ B 10.8	6 ○ C 12.01	7 * N 14.01	8 * O 16.00	9 * F 19.0	10 * Ne 20.2
11 ○ Na 23.0	12 ○ Mg 24.3											13 ○ Al 27.0	14 ○ Si 28.1	15 ○ P 31.0	16 ○ S 32.1	17 * Cl 35.5	18 * Ar 39.9
19 ○ K 39.1	20 ○ Ca 40.1	21 ○ Sc 45.0	22 ○ Ti 47.9	23 ○ V 50.9	24 ○ Cr 52.0	25 ○ Mn 54.9	26 ○ Fe 55.8	27 ○ Co 58.9	28 ○ Ni 58.7	29 ○ Cu 63.5	30 ○ Zn 65.4	31 † Ga 69.7	32 ○ Ge 72.6	33 ○ As 74.9	34 ○ Se 79.0	35 † Br 79.9	36 * Kr 83.8
37 ○ Rb 85.5	38 ○ Sr 87.6	39 ○ Y 88.9	40 ○ Zr 91.2	41 ○ Nb 92.9	42 ○ Mo 95.9	43 Δ Tc (99)	44 ○ Ru 101.1	45 ○ Rh 102.9	46 ○ Pd 106.4	47 ○ Ag 107.9	48 ○ Cd 112.4	49 ○ In 114.8	50 ○ Sn 118.7	51 ○ Sb 121.8	52 ○ Te 127.6	53 ○ I 126.9	54 * Xe 131.3
55 † Cs 132.9	56 ○ Ba 137.3	57 ○ La 138.9	58-71 LANTHANIDE Series	72 ○ Hf 178.5	73 ○ Ta 180.9	74 ○ W 183.9	75 ○ Re 186.2	76 ○ Os 190.2	77 ○ Ir 192.2	78 ○ Pt 195.1	79 ○ Au 197.0	80 † Hg 200.6	81 ○ Tl 204.4	82 ○ Pb 207.2	83 ○ Bi 209.0	84 ○ Po (210)	85 ○ At (210)	86 * Rn (222)
87 † Fr (223)	88 ○ Ra (226)	89 ○ Ac (227)	90-103 ACTINIDE Series	104 Δ Ku (260)	105 ?												

STATE AT 25°C

* gas
† liquid
○ solid
Δ synthetic

LANTHANIDE SERIES	58 ○ Ce 138.9	59 ○ Pr 140.1	60 ○ Nd 140.9	61 Δ Pm (145)	62 ○ Sm 150.4	63 ○ Eu 152.0	64 ○ Gd 157.2	65 ○ Tb 158.9	66 ○ Dy 162.5	67 ○ Ho 164.9	68 ○ Er 167.3	69 ○ Tm 168.9	70 ○ Yb 173.0	71 ○ Lu 175.0

ACTINIDE SERIES	90 ○ Th (227)	91 ○ Pa 232.0	92 ○ U (231)	93 Δ Np (237)	94 Δ Pu (242)	95 Δ Am (243)	96 Δ Cm (248)	97 Δ Bk (247)	98 Δ Cf (249)	99 Δ Es (254)	100 Δ Fm (253)	101 Δ Md (256)	102 Δ No (254)	103 Δ Lw (257)

Adapted by John E. Dyer

© Copyright 1973 by Marcelline A. Barron

All rights reserved. No part of this publication may be reproduced or transmitted in any form or by any means, electronic or mechanical, including photocopy, recording, or any information storage or retrieval system, without express permission in writing from the author.

Cover design and illustrations by Monica Barron.

Typescript prepared by Thomas J. Hadfield.

Printed in the United States of America
by Edwards Brothers Incorporated.

To my father and Father
...both of whom are in heaven.

PREFACE

To the Student

The growth of chemistry as a science is based upon two very different kinds of developments. First, experimental research provides us with the fundamental factual *information* which is the basis of all chemical knowledge. There is nothing more basic or more important to any science than the data which is obtained through research and experimentation in the laboratory. Secondly, chemists develop various *generalizations* by which they try to explain, interpret, and use the facts which have been discovered. Theories, principles and laws are examples of this process of generalization. Chemists, then, have two major and distinct activities: (1) discover facts about nature through experimentation, and (2) to formulate generalizations from this information. In your chemistry class, you will probably notice that the investigations emphasize these processes.

This book is especially concerned with the first activity—laboratory work. With fundamental chemical facts gained in the laboratory, your participation in discussion sessions can focus on formulating generalizations from pieces of data. With this twofold approach, you should be able to develop a genuine understanding of the true nature of chemistry.

You will gain in self-reliance by making each experiment a job well done. Familiarize yourself with each assigned experiment *before* coming to the laboratory. Prepare your data tables ahead of time so that you will only have to refer to your manual for instructions while working. Do your part when working with your partner; work together as a team, each person contributing something to the investigation. Use both ingenuity and common sense, for you will find that there are many opportunities for *both* logical and imaginative thinking.

Remember that your laboratory work is the core of your chemistry course. You, as an investigator, have a unique and challenging opportunity to observe many of the phenomena of chemistry. It is just from such experimentally-obtained observations that scientists have developed the principles that unify chemistry, and the theories which provide the current explanations. This is the scientific method in action.

Safety Considerations

In view of the many periods that you will be spending in the laboratory, it is *strongly* suggested that you familiarize yourself with the following basic safety rules:

(1) Safety goggles and laboratory aprons or coats must be worn at all times by all persons in the laboratory.
(2) Every laboratory should be equipped with a fire extinguisher, fire blanket, overhead shower, and first-aid supplies. Determine where these items are located in your laboratory and be sure that you know how to use them.
(3) Perform *only* those experiments that you have been specifically authorized to do.
(4) Notify your teacher about any accident, *no matter how minor*.

Acknowledgements

This book could not have become a reality without the encouragement and participation of others. Personal thanks are due the following people at Edwin Denby High School: Mr. Haze Moore, science department head, for his enthusiastic support during the formative stages of the manual; Mr. Thomas Vandenameele, science teacher, for his cheerful and conscientious suggestions during the testing of the material; and various laboratory assistants who helped prepare solutions and assemble equipment for use in the course of study.

I am indebted to all those who helped with the mechanics of preparing this manuscript for publication: my sister, Monica Barron, for the cover design and all the illustrations; and Mr. Thomas J. Hadfield for his meticulous typing of the manuscript. Their assistance with proofreading is also appreciated. Valuable editorial comments were provided by Monica Barron, Thomas Hadfield, and John O'Malley.

Not to be forgotten is the patient encouragement of family and friends during the genesis and development of this book. In large measure, their support has made possible the creation of a chemistry course that is hopefully relevant to the needs of today's high school students.

Lastly, but most importantly, I would like to thank the many students at Edwin Denby High School who have class-tested these materials. Their candid responses, criticisms, and obvious enthusiasm have been a source of optimism and inspiration. May they always continue to be surprised by the joy of discovery.

Marcelline A. Barron
Detroit, Michigan
July, 1973

CHEMISTRY: COPING WITH CHANGE CREATIVELY

As informed members of the world community,
we need skills in observing reality,

↓

in order to interpret how reality changes.

↓

Models help us to categorize and quantify change.

↓

Our environment is constantly undergoing
change. Ecochemistry helps us to form a
model of a changing universe.

↓

Change also affects our collective
everyday life -- nutrition.

↓

Can we technologically control the results
of the chemical changes we make, to avoid
technological backlash?
(Or: Does our technology control us?)

↓

Looking to the future: shock or sensibility?
Are we achieving better living through chemistry?

CONTENTS

As informed members of the world community, we need skills in observing reality,

A-1 How Inquisitive Are You? ? ? 1
A-2 Observing Evidence of Interaction 2
A-3 Observation and Theory 3
A-4 Observation: A Scientific Tool 4
A-5 Scientific Observation and Description 6
A-6 The Ups and Downs of Burning 7
A-7 Burning Rates of Candles 8
A-8 The Conservation of Matter 10
A-9 Applying Chemistry to Your Daily Life 11

in order to interpret how reality changes.

B-1 Properties of Matter: Chemical and Physical Changes (I) 15
B-2 Properties of Matter: Chemical and Physical Changes (II) 17
B-3 Mixtures and Compounds 19
B-4 Properties of Matter: Chemical and Physical Changes (III) 21
B-5 A Study of the Phase Change Between a Solid and a Liquid 23
B-6 Melting Point 26
B-7 Melting Behavior of Crystals Vs. Amorphous Solids 28
B-8 Distillation 30
B-9 Dry Distillation of Wood 32
B-10 Chemical Changes and Equations 33
B-11 Chemical Change: Reduction of Lead 36
B-12 Chemical Change: Corrosion of Iron 38
B-13 Allotropic Forms of Sulfur 40
B-14 The Rate of a Reaction 41
B-15 A Study of Reaction Rates 44
B-16 Catalysts 46
B-17 An Expanded Study of Reaction Rates 48
B-18 The Effect of the Amount of a Catalyst 51
B-19A Conversion of Starch to Glucose: Enzymes as Catalysts 53

Models help us to categorize and quantify change.

C-1 Diffusion of Gases 56
C-2 Models of Some Molecules 57
C-3 The Preparation and Investigation of Two Isomers 60
C-4 Molecular Layers 62
C-5 The Preparation and Study of Crystals 64
C-6 An Investigation of the Packing of Atoms or Ions in Crystals 67
C-7 Determining the Size of Objects by Collisions 69
C-8 Creating an Electron Charge Cloud Analogy 71
C-9 Electron Probability Map Vs. Electron Path 73
C-10 A Half Life Model of Nuclear Chemistry 73
C-11A Collecting Radioisotopes from the Air 76

Our environment is constantly undergoing change. Ecochemistry helps us to form a model of a changing universe.

D-1 Solids in the Air 78
D-2 The Effect of Air Pollution on Visibility 79
D-3 The Composition of Air 81
D-4 Simplified Airborne Solid Sampling 84
D-5 Destructive Distillation of Leaves 86
D-6 The Properties of Sulfur Compounds 87
D-7 Preparation and Properties of Carbon Dioxide 91
D-8 Three Oxides of Nitrogen 94
D-9 Lead and its Compounds 95
D-10 Some Problems of Incineration 97
D-10A Total Acids in Air 98
D-11 Analysis of Soil for Some Common Constituents 100
D-12 Mineral Content of a Fertilizer 103
D-13 The Analysis of Substances Contained in Plant Tissue 106
D-14 Chemical Properties of Water 109
D-15 Solutions and Suspensions 113
D-16 A Quantitative Study of the Effect of Temperature on Solubility 116
D-17 Solubility 117
D-18 A Study of Hard Water 120
D-19 Comparison of Various Consumer Washing Products 122
D-20 Testing Water Samples for Dissolved Oxygen 126
D-21 A Study of Some Impurities in Water Samples 128
D-22 A Study of pH, Indicators, and Alkalinity of Water Samples 131
D-23 Water Treatment and Surface Chemistry 135
D-24.1A The Study and Identification of Certain Metallic Ions (I) 138
D-24.2A The Study and Identification of Certain Metallic Ions (II) 140

Change also affects our collective everyday life— nutrition.

E-1 The Preparation of Some Organic Compounds 143
E-2 Qualitative Tests for Some Organic Molecules in Common Foods 145
E-3 Organic Macromolecules—Proteins 147
E-4 A Study of Carbohydrates and Their Reactions 149
E-5 The Digestion of Starch 152
E-6 Consumer Food Test I: Butter and Margarine 154
E-7 Consumer Food Test II: Caffeine 157
E-8 Consumer Food Test III: Synthetic Dyes 159
E-9 Determination of Citric Acid and Vitamin C in Fruit Juices 162
E-10 A Study of the pH of Common Foods 164

Laboratory Equipment: Glass and Porcelain
Laboratory Equipment: Metal, Plactic and Rubber

EXPERIMENT A-1: HOW INQUISITIVE ARE YOU? ? ?

INTRODUCTION

The art of thorough and penetrating analysis is, for many scientists, a learned skill. The diverse observations which are revealed by a given reaction may require a variety of manipulations, a greater than average amount of originality, and the courage to proceed with some steps which may seem unorthodox. However, the most important attitude in science is the spirit of experimentation.

MATERIALS

Beaker; a wooden splint or stirring rod; powder A; powder B; magnifying glasses or hand lenses.

INSTRUCTIONS

Obtain a sample of powder A and powder B in your beaker. Observe as much about the two powders as you can. *Caution: Do not taste or heat them.* Write down your observations. After you are done, take your beaker to a collection area in the laboratory. Take a look at the contents in the beakers of the other students. Compare them with yours. Record all of your observations.

OBSERVATIONS

1.
2.
3.
4.
5.
6.
7.
8.
9.
10.
11.
12.
13.
14.
15.
etc.

EXPERIMENT A-2: OBSERVING EVIDENCE OF INTERACTION

INTRODUCTION

This experiment provides another opportunity for careful observation. Try to think of conditions that might make a difference in what you will observe. Be alert to questions that come to mind and make your observations as complete as possible.

MATERIALS

Blue crystals; beaker; distilled water; aluminum foil square; stirring rod.

INSTRUCTIONS

Add about two level teaspoonfuls of blue crystals to a beaker which is 3/4 full of distilled water.

Allow the system of water and crystals to stand a few minutes. Record your observations.

Stir the water until the crystals dissolve. After the crystals dissolve, place a piece of loosely rolled aluminum foil into the liquid. Allow the foil to remain in the solution for 10-15 minutes. Then remove and examine it closely with hand lenses.

After listing your observations, write down all the questions that occurred to you during this experiment.

OBSERVATIONS

1.

2.

3.

4.

5.

etc.

QUESTIONS

1.

2.

3.

4.

5.

etc.

EXPERIMENT A-3: OBSERVATION AND THEORY

INTRODUCTION

Chemistry deals with the changes which matter undergoes. Since the changes are on a molecular level, we cannot directly observe how they occur. What we usually observe are the differences between the properties of the initial matter (reactants) and the properties of the final matter (products). In the laboratory, experiments are conducted in which the initial and final properties of matter are studied and compared. Afterwards the chemist attempts to develop a theory to explain his observations of the changes that took place.

A sealed box containing one or more unknown solid objects will serve as the matter which you will be observing in this experiment.

MATERIALS

Unsealed cardboard box or shoe box; sealed box with unknown object enclosed; ruler; platform balance; magnet.

INSTRUCTIONS

Examine the unsealed box and record its code number. Weigh the empty box to the nearest 0.1 gram. Record its weight on the data sheet.

Examine the sealed box and record its code number. Determine its dimensions to the nearest centimeter. Weigh the sealed box. Record its weight.

Manipulate (shake, tilt, etc.) the sealed box. Use a magnet to study the contents of the box. For each manipulation listen carefully to the sound inside and record your observations. After each manipulation, try to form conclusions about the object inside the box. Record these conclusions on the data table.

DATA

1. Code number of unsealed box _____
2. Weight of unsealed box = _____ grams
3. Code number of sealed box _____
4. Width of sealed box = _____ centimeters
5. Length of sealed box = _____ centimeters
6. Height of sealed box = _____ centimeters
7. Weight of sealed box = _____ grams
8. Difference in weight between sealed and unsealed boxes = ____ grams

OPERATIONS TABLE

Manipulation	Observation	Conclusion
1. Gentle Shaking		
2. Tilting		
3. Magnet Testing		
4. Dropping (1 ft.)		
5. Sliding		
6. Rocking		
7. Vigorous Shaking		
8.		
9.		

What do you think your unknown looks like? Give reasons for your theory. Draw a sketch of your unknown.

EXPERIMENT A-4: OBSERVATION: A SCIENTIFIC TOOL

INTRODUCTION

What is chemistry? Like physics and biology, it is a basic science which deals with matter. It is different from physics and biology because it is a study of the properties, composition, and structure of matter, of the changes that occur in matter, and of the energy absorbed or released during these changes.

We use our senses to hear, see, smell, taste, and feel chemical reactions. In the modern chemistry laboratory, each of these senses may be supplemented by other devices to help us make more precise and accurate observations, such as balances and magnifying glasses.

MATERIALS

Solutions A, B, and C; test tubes; china marker; distilled water; small piece of metal; test tube rack.

INSTRUCTIONS

Obtain three test tubes and a supply of the solutions marked A, B, and C. Label three test tubes (A,B,C). Fill the test tubes one-third full so that you have a test tube one-third full with Solution A, another tube one-third full with Solution B, and a third tube one-third full with Solution C. Slowly pour the contents of test tube A into test tube B. Observe and record the results.

Refill test tube A with Solution A. Pour the contents of test tube A into test tube C. Observe and record the results. Empty the test tubes and wash them first with tap water and then rinse them with distilled water. Refill test tubes B and C. Pour the contents of test tube B into test tube C. Observe and record the results. Empty the test tubes and wash them again just as you did above.

Finally, once again fill the test tubes one-third full of Solutions A, B, and C. Slowly drop a small piece of the metal provided for you by your teacher into each tube. Spend a few minutes observing what is occurring to the metal in the liquids. Record all of your observations.

OBSERVATIONS

	Solution B	Solution C
Solution A		
Solution B		

	Solution A	Solution B	Solution C
Metal			

EXPERIMENT A-5: SCIENTIFIC OBSERVATION AND DESCRIPTION

INTRODUCTION

Everyone thinks of himself or herself as a good observer. Yet there is much more to observation than what is immediately obvious. Concentration, alertness to detail, ingenuity, and often just plain patience are necessary for meaningful observation. It even takes practice. A good scientist pays close attention to the discovery and control of experimentally important conditions.

MATERIALS

Candle; matches; liner for underneath candle; ruler.

INSTRUCTIONS

First examine the candle carefully. Record all of the observations which you make. Place a liner underneath the candle, then light the candle and record as many observations as you can about the burning candle.

After making all observations, check to be sure that no candle wax was spilt on the laboratory table. If so, remove the wax immediately. *Do not wash excess wax down the drain. Wax should be put into the chemical crock.*

OBSERVATIONS

1.
2.
3.
4.
5.
6.
7.
8.
9.
10.
11.
12.
13.
14.

etc.

Write a description of a burning candle, based upon your list of observations.

EXPERIMENT A-6: THE UPS AND DOWNS OF BURNING

INTRODUCTION

This experiment will attempt to study the process of burning or combustion. What happens during combustion? Will a burning candle gain or lose weight during the process of combustion? Maybe it will stay the same. Does this happen when other substances burn?

MATERIALS

Candles; matches; meter stick; ringstand with ring clamped on it; flat object; liner for candle; crucible; asbestos square; magnesium strip.

INSTRUCTIONS

Balance a meter stick horizontally on the clamp of a ring stand as indicated in Figure A-6.

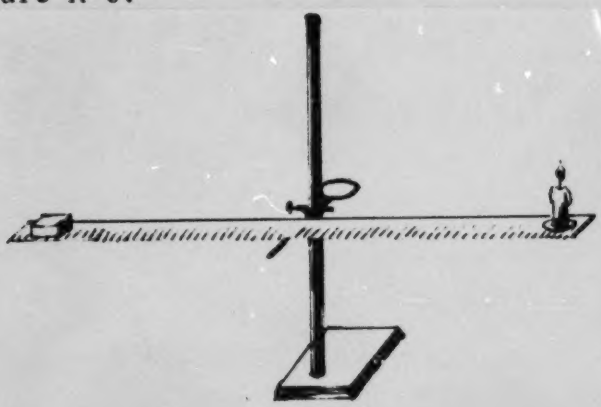


Figure A-6.

Place a candle and a liner on one end of the meter stick. At the other end place a flat object such as a box of paper clips, aneraser, some rubber stoppers, etc. (use your imagination!). Position the flat object so that the whole system is balanced.

Light the candle. As it burns, speculate as to what will happen. Record exactly what did happen. Be sure that no wax is allowed to drip off the candle liner onto the table.

Remove the candle and flat object from the meter stick. Measure out a 50 cm. length of magnesium ribbon. Wind it into a loose coil which will fit into a crucible with a short end extending out to act as a fuse. Place the crucible with asbestos liner and magnesium ribbon in it on the meter stick where the candle was before. Place the flat object on the other end and balance the system again.

Speculate what will happen to the balance of the system when the magnesium is lighted. *Warning: Do not look directly at the magnesium when it is burning. The bright light could be harmful to your eyes if watched for more than a few seconds.* Carefully ignite the magnesium with a Bunsen burner. Stand back from the system and observe what is happening to the system. After the crucible cools, examine the contents and record your observations.

When cleaning up after this experiment, be sure to place the residue from the crucible and the candle in the proper containers.

DATA

1. What happened to the system when the candle burned? (INCLUDE SKETCH).
2. What happened to the system when the magnesium burned? (INCLUDE SKETCH).
3. What did the contents of the crucible resemble after burning?
4. Was there any difference in the behavior of these two systems when burning occurred?
5. Check your results with those of other students. How does your data compare with theirs?

EXPERIMENT A-7: BURNING RATES OF CANDLES

A candle flame has three zones of combustion: an inner zone adjacent to the wick where the wax is vaporized; a middle zone of partial burning where small carbon particles are released from the hydrocarbons in the wax; and an outer zone where wax combines with oxygen to yield water vapor and carbon dioxide.

The candle wick is a porous structure which sucks up the melted wax by capillary action and carries it to the inner layer of the flame where it is vaporized. For proper burning the supply of melted wax must equal but not exceed the demand; that is, the wax must melt only fast enough for an adequate supply to move up the wick. Therefore, the candle diameter determines the amount of melted wax available, and the wick diameter determines the amount which can be carried to the center of the flame.

MATERIALS

Candle; liner for candle; matches; compass; cardboard; scissors; ruler.

INSTRUCTIONS

Use the compass and prepare some cardboard collars for your candle of various widths ranging from 2 in. to 6 in. in *diameter.* Choose a 1/2 inch interval on your compass, for example, 1 in., 1 1/2 in., 2 in., etc.

Cut the proper hole in the center of the collars so that the candle will fit into it. Place the collars near the top of the candle, that is near the lip of the candle.

Determine whether the width of the collar has any effect upon the burning rate of the candle. Make your observations and record them. Is there any relationship between the increased width of collar on the candle and the burning behavior of the candle?

DATA

Width of Collar	Observations
2 inches	
3 inches	
4 inches	
5 inches	
6 inches	

These candles and their collars are meant to simulate candles with increasing diameters and how the air currents play a role in the rate of burning. What conclusions can you come to regarding the relationship between increased candle diameter and rate of combustion?

EXPERIMENT A-8: THE CONSERVATION OF MATTER

INTRODUCTION

Is there any relationship between the reactants and the products of a chemical reaction that is not readily observable and might escape notice? This experiment will examine several chemical reactions from this viewpoint.

MATERIALS

250 ml Erlenmeyer flask and solid rubber stopper to fit; small test tubes; balance; dilute hydrochloric acid; solutions of sodium sulfate, potassium chromate, silver nitrate, barium chloride and lead nitrate; distilled water; graduated cylinder.

INSTRUCTIONS

Obtain a 250 ml Erlenmeyer flask with a tightly fitting rubber stopper. Fill the test tube three-quarters full of the silver nitrate solution. *(Caution: Avoid spilling silver nitrate on your hands, since it will stain the skin dark brown. Rinse immediately with water and a sodium chloride solution if spilling occurs. Small amounts are not injurious, and the discoloration wears off after several days.)*

Place 20 ml of dilute hydrochloric acid (HCl) into the flask; lower the test tube carefully into the flask and stopper the flask. See Figure A-8.

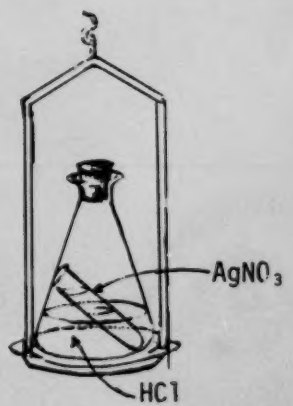


Figure A-8.

Carefully weigh the flask and its contents. Make certain that the outside of the flask is *dry* before you proceed with the weighing.

Next, invert the flask several times so that the contents of the tube will mix thoroughly with that of the flask. Observe the reaction that takes place and the product that is formed. Record your observations. Once more set the flask on the balance and weigh it. Record the weight.

Clean the equipment thoroughly and repeat the procedure described above, this time filling the test tube three-quarters full of barium chloride solution and the flask with 20 ml of sodium sulfate solution. Record the results of the mixing and weighing.

Again, repeat the procedure described above, this time placing lead nitrate solution in the test tube, and potassium chromate in the flask. Record your observations after mixing and weighing.

DATA

Reactants	Weight Before	Weight After	Weight Change	Observations
Silver Nitrate and Hydrochloric Acid				
Barium Chloride and Sodium Sulfate				
Lead Nitrate and Potassium Chromate				

What were the obvious changes in properties that indicated a chemical reaction must have taken place in the three systems above?

What relationship between products and reactants might have escaped your notice if closed flasks and balance were not used?

The Law of Conservation of Matter states that, under normal conditions, matter can neither be created nor destroyed. Do you believe that you have verified this law? State your evidence to support your answer.

EXPERIMENT A-9: APPLYING CHEMISTRY TO YOUR DAILY LIFE

INTRODUCTION

The activities in this experiment are intended to acquaint you with some of the ways in which chemistry enters your daily life. From time to time you will acquire new skills and laboratory techniques that can be useful in understanding the chemical aspects of materials around you.

SECTION I: Estimating the Volume of a Container

MATERIALS

Various bottles, jars and tin cans of different shapes and sizes; water; graduated cylinders of various sizes.

INSTRUCTIONS

At times it is desirable to be able to estimate with some accuracy the volume of a container. This is a skill which can be acquired only by practice.

Obtain a number of different types of containers. Make your own estimate of the capacity in milliliters of a container. Fill the container with water; then pour this water into a graduated cylinder marked in milliliters. Opposite your estimated capacity, record the capacity of the container as you measured it. Repeat this procedure for each of the containers you have chosen.

SECTION II: Percentage of Water in Canned Foods

MATERIALS

Two or three different brands of one type of canned food; graduated cylinder (100 ml); balance; 250 ml beaker; can openers.

INSTRUCTIONS

The amount of water as related to solid matter in canned foods may vary from one brand to another. Select two brands of a canned food. The cans should be of the same size for a fair comparison. Carefully open each can without spilling any of the liquid.

Carefully empty one of the cans into a glass funnel provided. Catch the liquid in a large graduated cylinder. After allowing plenty of drainage time, record the volume of liquid in the brand of food. Since one milliliter of water weighs one gram, what was the weight of liquid in the can? Record this weight on the data table.

Now determine the weight of the food by weighing it in a beaker that has been carefully weighed in advance. Record the weight of the food on the data table.

Repeat the above procedures with each brand of canned food to be tested and record the data on the table.

SECTION III: Percentage of Soluble Matter in Tea

MATERIALS

Tea leaves of three different brands; large beaker; balance; glass funnel; filter paper.

INSTRUCTIONS

A satisfactory brand of tea should consist of at least 40% (by weight) of soluble matter. Using simple laboratory techniques you may test several brands of tea.

Heat 250 ml of water in a beaker. While the water is heating, weigh out accurately 1.0 gram of tea leaves.

Place the weighed tea in a folded weighed filter paper in a glass funnel. When the water is heated to boiling, pour enough of it over the tea sample to cover it. As the water filters through the paper, keep adding more– a small volume at a time. When the liquid coming through the funnel (filtrate) begins to come through uncolored, all of the soluble matter has been removed from the tea leaves.

Dry the remaining tea leaves by leaving them exposed to the air until a later laboratory period in your lab drawer, in an evaporating dish on a hot plate, or in a drying oven (see hint below). In the data table record the data for each sample tested and compute the percentage of soluble matter as shown.

Hint: If a hot plate is used to dry the tea leaves, be careful not to burn them or the filter paper and be sure that the leaves are cooled to room temperature before weighing them. This precaution should also be kept in mind when using a drying oven as well.

DATA

SECTION I

Type of Container	Estimated Capacity	Measured Capacity
1.		
2.		
3.		
4.		
5.		
6.		

SECTION II

Weight of empty beaker = _____.

Name of Brand	a=Grams of water	b=Grams of solid	% water = $\frac{a}{a+b} \times 100$
1.			
2.			
3.			

Which is the most economical brand to purchase?_____

SECTION III

Name of Brand	a=Original weight of tea (1.0 gm)	b=Weight after extracting and drying*	$c=a-b$ weight of soluble matter	% soluble matter $c/a \times 100$
1.				
2.				
3.				

* Remember to subtract the weight of the filter paper.

Which brand of tea seems to be the most economical to use?_____

EXPERIMENT B-1: PROPERTIES OF MATTER
CHEMICAL AND PHYSICAL CHANGES (I)

INTRODUCTION

Chemical and physical *properties* must not be confused with chemical and physical *changes*. Some physical properties are: color, odor, density, hardness, structure, solubility, melting point, and boiling point. Chemical properties are determined by the reaction of a substance with other substances. An example of a chemical property would be the potential an atom has to lose one of its electrons and form an ion. This is called by chemists the ionization energy of the atom.

Physical change refers to a transformation of a substance in which the substance itself does not change. Chemical change refers to the production of new substances which have physical and chemical properties different from the original substance.

MATERIALS

Copper wire or foil; zinc (Zn) strips; magnesium (Mg) ribbon; forceps; dilute hydrochloric acid (HCl); concentrated nitric acid (HNO_3); solid sodium carbonate; distilled water; porcelain dishes; solution of calcium chloride ($CaCl_2$); filter paper; funnel; 150 ml beaker.

INSTRUCTIONS

Examine a clean piece of copper wire or copper foil and note its physical properties. Hold the piece of copper with the forceps in the outer flame of the Bunsen burner until the copper is red-hot. Remove the copper from the flame and observe it while it cools. Compare the physical properties of the product on the surface of the copper with the properties of the original copper.

Repeat the procedure with a piece of zinc and then with a piece of magnesium ribbon. *Do not look directly at the magnesium in the flame. The brightness of the flame could harm the eyes.* Record in the data table the properties of each metal before heating and the properties of any new substance produced by the heating. In each case state whether a chemical or a physical change was brought about with the application of heat to the metal.

Place the piece of zinc, a clean piece of copper, and a piece of magnesium ribbon in separate test tubes and add 4 ml of dilute hydrochloric acid (HCl) to each. Note any evidence that these metals are reacting with the acid. What results do you observe? Record them on the data table. Repeat this part of the experiment, adding a few drops of concentrated nitric acid (HNO_3) instead of hydrochloric acid. You may find it necessary to use the fume hood.

Describe what you have observed. After making your observations, add plenty of water to the test tubes. Place the copper, zinc, and magnesium in the proper disposal containers.

Obtain about 2 grams of sodium carbonate (Na_2CO_3) and note its physical properties. Dissolve it in 30 ml of distilled water, warming the water to hasten solution. Divide this solution into two equal portions and proceed as follows:

(1) Evaporate one portion to dryness with a low flame in a porcelain dish and compare the residue with the original substance.

(2) To the second portion add dilute hydrochloric acid (HCl), a few drops at a time, while stirring, until no further action is observed. Then evaporate to dryness in a porcelain dish. Examine the residue and compare its appearance with that of the original solid.

DATA

Substance	Physical Properties		Physical Change		Physical Change	
	Before Heating	After Heating	Yes	No	Yes	No

Substance	Reaction with Dilute Hydrochloric Acid	Reaction with Concentrated Nitric Acid

Substance	Observations
Sodium Carbonate	
Residue (1)	
Residue (2)	

EXPERIMENT B-2: PROPERTIES OF MATTER
CHEMICAL AND PHYSICAL CHANGES (II)

INTRODUCTION

In this experiment you will witness many transformations of matter. You must decide whether these transformations are physical or chemical changes and be able to give supporting evidence for your decisions.

MATERIALS

Wood splints; solid sodium chloride (NaCl); distilled water; copper turnings; dilute nitric acid (HNO_3); solid ammonium dichromate ($(NH_4)_2Cr_2O_7$); calcium carbide (Ca_2C); solutions of barium nitrate ($Ba(NO_3)_2$), sodium chromate (Na_2CrO_4), iron (III) chloride ($FeCl_3$), potassium thiocyanate (KSCN), silver nitrate ($AgNO_3$); liquid soap or detergent; forceps; eyedropper; test tubes; crucible; glass slide; balance; evaporating dish; ruler; asbestos pad; test tube holder; triangle to fit crucible.

INSTRUCTIONS

Classify each of the following transformations as a physical or chemical change and record your supporting evidence for your choice on the data sheet.

Obtain a wood splint and break it into small pieces. Place several pieces in a test tube and heat strongly over a bunsen burner for several minutes. Record the results.

Place 0.5 gram of sodium chloride (NaCl) into a beaker and add distilled water until the solid dissolves. Place two drops of the resulting solution on a glass slide. Hold the slide with forceps and pass it back and forth over a *low* flame. After the liquid has evaporated, taste the residue. *Caution: Never taste chemicals unless you are specifically instructed to do so.* Record the results.

Obtain a small amount of copper turnings from the supply. Roll them into a loose ball, place it in a dry crucible and weigh the crucible and contents to the nearest 0.01 gram. (The crucible should be dry before you weigh it. To dry the crucible, heat it strongly for 2 or 3 minutes and then let it *cool completely.*) Heat the metal in the crucible, all of which is setting in a wire triangle (see Figure B-2) over a hot flame for five minutes, cool, and reweigh. Record your data and observations.

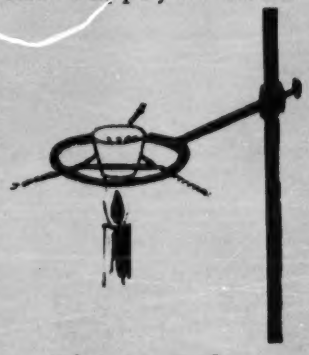


Figure B-2.

Obtain three clean large test tubes and mix the following solutions:

(1) To 5 drops of barium nitrate ($Ba(NO_3)_2$), add 2 to 3 drops of sodium chromate (Na_2CrO_4). Observe and record your observations.

(2) To 5 drops of iron (III) chloride ($FeCl_3$), add 3 drops of potassium thiocyanate (KSCN). Observe and record your observations.

(3) To 5 drops of iron (III) chloride ($FeCl_3$), add 2 drops of silver nitrate ($AgNO_3$). Observe and record your observations.

With your forceps, obtain a small piece of calcium carbide (Ca_2C). Place it in an evaporating dish containg 20 ml of distilled water and 3 drops of liquid soap. During the reaction, ignite the bubbles with a lighted splint.

In the bottom of a large test tube, place ammonium dichromate ($(NH_4)_2Cr_2O_7$) to a height of one centimeter and heat until a reaction starts. Remove the test tube from the flame and allow the reaction to go to completion. *Caution: Do not inhale the fumes.*

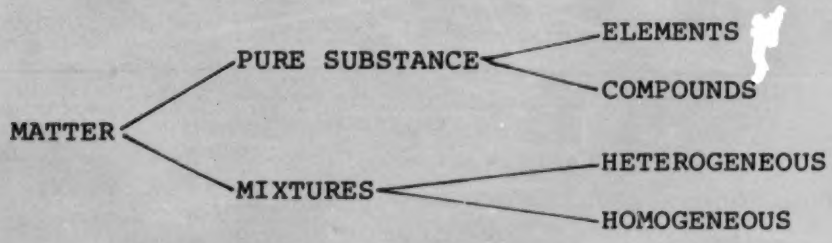
DATA

Exercise	Type of Change	Supporting Evidence
Heating Wood		
Evaporation of sodium chloride solution		
Heating copper turnings		
Reaction of barium nitrate with sodium chromate		
Reaction of iron chloride with potassium thiocyanate		
Reaction of iron chloride with silver nitrate		
Reaction of calcium carbide with water		
Heating ammonium dichromate		

EXPERIMENT B-3: MIXTURES AND COMPOUNDS

INTRODUCTION

The classification or organization of matter is of great importance in the study of chemistry. One possible classification is as follows:



MATERIALS

Sand, table salt; distilled water; 150 ml beaker; funnel; filter paper; evaporating dish; iron filings; flowers of sulfur or powdered sulfur; magnet; carbon disulfide (CS_2); mercuric oxide (HgO); splints; matches; balance; graduated cylinder (50 ml); mortar and pestle.

INSTRUCTIONS

Mix a test tube filled one quarter full with sand with an equal portion of table salt on paper. Place the mixture in a beaker and add about 25 ml of distilled water. Stir thoroughly. Filter the liquid with a funnel and filter paper. Taste the residue on the filter paper. Boil the filtrate (the liquid which passes through the filter paper) in an evaporating dish until the water has evaporated and taste the solid remaining in the dish. *(Reminder: Never taste chemicals unless you are specifically instructed to do so.)*

Mix a quarter test tube of iron filings with an equal portion of flowers of sulfur. Divide the mixture into two parts. Dip one end of a bar magnet into one portion, and then tap that end against your hand. Is this a physical or chemical method of separation? Record your observations.

Before beginning this section, *check to be certain that all flames at your lab table are extinguished. Be sure that all flames are out.* Place a little of the second portion of the mixture of iron filings and flowers of sulfur in about 5 ml of carbon disulfide in a beaker. Stir thoroughly and filter into an evaporating dish. Set the dish in the hood or near an open window. When the carbon disulfide has evaporated, describe the deposit in the dish. Was this a physical or chemical method of separation?

Heat 2 grams of red mercuric oxide in a Pyrex test tube. Insert a glowing splint into the tube after heating for one minute. After about 5 minutes of heating describe the inner surface of the upper

part of the tube. What happens? Is this a physical or chemical change?

Mix thoroughly on paper 7 grams of iron filings and 4 grams of flowers of sulfur. Place the mixture in a Pyrex test tube. Heat the end of the tube until a glow appears; then remove it from the flame. Heat the materials for several more minutes. Break the hot tube by holding it in cool water. Be careful for the substance in the tube is still hot. Bring a magnet near a particle of the substance. Crush a little of the material from the tube in a mortar and pestle. When it has cooled, add about 5 ml of carbon disulfide. Check to make sure all flames are *out*. Stir the mixture several minutes; then filter the liquid into a dish and let it evaporate. Observe.

DATA

Sand-Salt Mixture

Is there any salt in the residue?

What is the taste of the solid remaining in the evaporating dish after evaporation?

Were physical or chemical methods used in the separation of this mixture?

Iron-Sulfur Mixture

Which substance in the mixture is removed by the magnet?

Is this a physical or chemical method of separation?

Describe the deposit in the dish after the carbon disulfide evaporated.

Decomposing a Compound

Describe the inner surface of the upper part of the tube in which you heated the mercuric oxide.

What is the result of the glowing splint?

Into what two substances does mercuric oxide decompose?

Do either of these substances resemble the mercuric oxide?

Was the separation a physical or chemical change?

Formation of a Compound

Can you distinguish any particles of iron or sulfur in the mass after heating and breaking the glass?

After using the carbon disulfide and allowing it to evaporate, is there a deposit of sulfur in the dish?

Can iron and sulfur in chemical combination be separated by physical means?

EXPERIMENT B-4: PROPERTIES OF MATTER
CHEMICAL AND PHYSICAL CHANGES (III)

INTRODUCTION

In this experiment you will examine two compounds to determine the effect that heat has on them. Both of the compounds belong to a group that is named "organic compounds."

MATERIALS

Sugar ($C_{12}H_{22}O_{11}$); naphthalene; test tubes; charcoal; evaporating dish; eyedropper or pipette; glass funnel; filter paper; litmus paper; beaker (150 ml); dilute hydrochloric acid (HCl).

INSTRUCTIONS

Place a teaspoon of sugar in a test tube. Heat the tube gently and observe the stages of decomposition.

Place a teaspoon of naphthalene in a large test tube. Observe its crystalline form and the odor of the substance. Heat gently until it melts and observe its color. Raise the temperature until the naphthalene boils. Collect the condensed product in a test tube as is shown in Figure B-4.1 and compare it to the solid that you originally started with. Record your observations. (*Hint:* Use benzene to clean the test tubes.)

Place an evaporating dish containing 2 grams of sugar on a piece of wire gauze on a ring and ring stand arrangement as shown in Figure B-4.2. Invert a glass funnel over the sugar in the evaporating dish and light the Bunsen burner to produce a low-to-moderate flame.

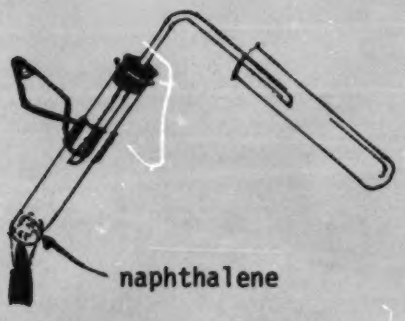


Figure B-4.1.

Figure B-4.2.

As the sugar melts and eventually bubbles, observe the condensation of liquid on the inner walls of the funnel. When enough droplets have formed there, remove the funnel and soak up the liquid with some litmus paper (blue and red). Record the results.

Allow the sugar to bubble slowly until all the liquid has been driven off. Continue to heat until the sugar has changed color completely from white to black. Turn off the burner and allow the residue in the evaporating dish to cool.

Obtain a small bit of charcoal or finely divided carbon from the supply. Put the charcoal or finely divided carbon in a beaker of cold tap water. Scrape a small amount of the residue from the evaporating dish into another beaker of cold tap water. Observe the solubility or lack of it in the case of both substances.

Using an eyedropper, put a few drops of dilute hydrochloric acid (HCl) on both the commercially prepared charcoal and the residue in the evaporating dish. Observe the results.

DATA

Melting Behavior	
Sugar	Naphthalene

Boiling Behavior	
Sugar	Naphthalene

Results of litmus test:

Solubility Results			
Water		Dilute Hydrochloric Acid	
Commercial Charcoal	Prepared Charcoal	Commercial Charcoal	Prepared Charcoal

EXPERIMENT B-5: A STUDY OF THE PHASE CHANGE BETWEEN A SOLID AND A LIQUID

INTRODUCTION

A solid moth repellant, paradichlorobenzene, will be heated slightly above its melting temperature in this experiment. Temperature data will be obtained by noting the temperature at designated time intervals as the liquid cools and solidifies. The solid will then be reheated and melted, and again temperature data will be obtained at designated time intervals during the heating.

MATERIALS

Solid paradichlorobenzene; two centigrade thermometers; test tube; test tube holder; ring and ring stand; 250 ml beaker; burner; matches; timer; wire gauze; clamp.

INSTRUCTIONS

Fill a 250 ml beaker three-fourths full of tap water at about room temperature (below 30°C). Place the beaker on the base of your ring stand.

Slowly raise the temperature of about 15 grams of paradichlorobenzene in a test tube until it melts. Heat the test tube with the Bunsen burner adjusted to a low flame. Continuously move the test tube back and forth through the flame as the liquid will spurt out of the tube if you keep it in one spot in the flame! Place a thermometer in the liquid and continue heating until the temperature is between 65°C and 70°C.

Clamp the test tube containing the melted paradichlorobenzene into position above the beaker of water. Place your watch or situate

yourself so that one partner can read the time every 30 seconds and be the recorder of the temperature readings.

When all is ready, check the temperature of the paradichlorobenzene and record it to the nearest division on your thermometer. On signal from the recorder, immediately the other partner should immerse the lower half of the test tube into the water bath and clamp the test tube in position. Hold the thermometer off the bottom and the sides of the test tube so that it will become imbedded in that position when the paradichlorobenzene solidifies. Record the temperature every 30 seconds until a temperature of 35°C is reached. Record all observations especially when the solidification starts and when it is complete.

You may wish to switch duties at this point. Raise the test tube out of the water bath and place the beaker, three-fourths full of water, on an iron ring and wire gauze. Heat the water bath to approximately 70°C. When this temperature is reached, turn off the burner but leave it in position.

Record the temperature of the solidified paradichlorobenzene to the nearest division on your thermometer. With a second thermometer measure the temperature of the hot water bath to the nearest degree. On the signal from the recorder, immerse the lower half of the test tube into the water bath until the water level is above the level of the paradichlorobenzene. Record the temperature of the paradichlorobenzene each 30 seconds, noting when melting starts and is completed.

As soon as the solid is free from the walls of the test tube, move the thermometer gently up and down. Occasionally note the temperature of the water bath with the second thermometer. If the water temperature drops below 60°C before the solid is melted, be prepared to warm it with the burner flame to keep the temperature between 60°C and 65°C. Continue to move the thermometer up and down in the test tube and record the temperatures until the temperature of the paradichlorobenzene is about 60°C. Then remove and wipe off the thermometer from the liquid paradichlorobenzene.

DATA

Solidification Data		
Time	Temp. (°C)	Observations
0 sec		
30 sec		
1 min		
1½ min		
2 min		
2½ min		
3 min		
3½ min		
4 min		
4½ min		
5 min		
5½ min		
6 min		
6½ min		
7 min		
7½ min		
8 min		

Melting Data		
Time	Temp. (°C)	Observations
0 sec		
30 sec		
1 min		
1½ min		
2 min		
2½ min		
3 min		
3½ min		
4 min		
4½ min		
5 min		
5½ min		
6 min		
6½ min		
7 min		
7½ min		
8 min		

After you obtain some graph paper, plot your data in the following manner:

On the first graph, the time from 0 sec. to 8 min. should be marked off along the horizontal axis and the temperature should be marked off on the vertical axis from 0°C to 70°C (see Figure B-5). This graph should be labeled "Cooling Curve."

On the second graph the time and temperature axes will be the same, but this graph should be labeled "Heating Curve." After plotting all the points from the data tables, be sure to connect all your points with a nice smooth curved line.

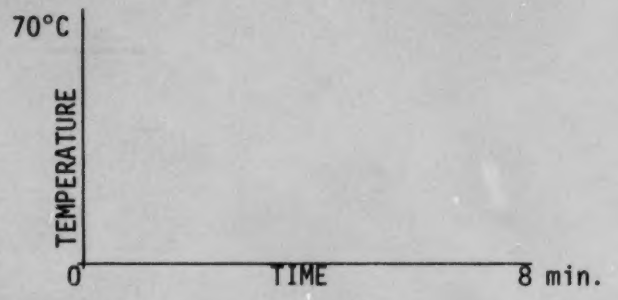


Figure B-5.

EXPERIMENT B-6: MELTING POINT

INTRODUCTION

The determination of a melting point is a technique employed often by the chemist. A melting point may be defined as the temperature at which the liquid and solid forms of a compound exist in equilibrium with each other. In ordinary practice a melting-point range is recorded, which means the temperature from which the compound starts to liquefy to the point where it is completely liquid. Many very pure compounds melt over 0.5°C range or less. The melting-point range of a compound of ordinary purity is 1-2°C. The value obtained for a compound may, therefore, provide some indication of its purity. It should be noted that some compounds, even though they may be exceptionally pure, have wide melting ranges. If a substance is not pure (it is a mixture) it will not melt sharply.

MATERIALS

Oil or paraffin; 250 ml beaker or flask; capillary tubes; centigrade thermometer (with a range up to 200°C); glass stirrer; ring and ring stand; rubber tubing strip (2 mm); solid naphthalene; benzoic acid; and salicylic acid crystals or powder.

INSTRUCTIONS

Fill a 250 ml beaker with oil or paraffin and place on ring attached to a ring stand with wire gauze between the container and the flame.

Capillary tubes will be used to hold the sample against the thermometer bulb. You will be determining its melting point by using an apparatus as shown in Figure B-6.1. First, the sample must be

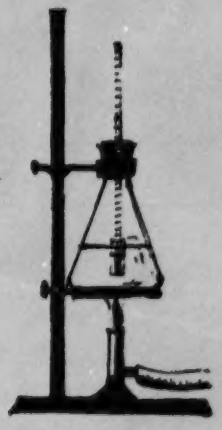


Figure B-6.1.

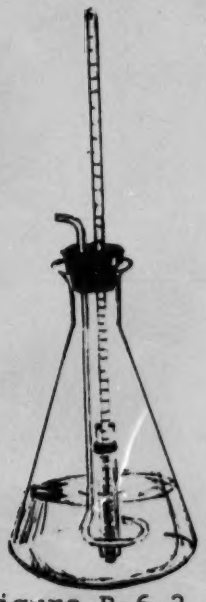


Figure B-6.2.

dry and in a finely divided condition so that it will go into the capillary tube. If both ends of the tube are open, seal one end by passing it through a flame. Form a small pile of the solid and press the open end of the tube into it. Tap the closed end of the tube against the desk in order to get the compound to drop to the bottom of the tube. If the crystals stick in the upper part of the tube, attempt to force them down by dropping the capilary tube through some glass or plastic tubing. Fill the capillary tube to a height of 5-6 mm, then place it against the side of the thermometer with the compound close to the mercury bulb as shown in Figure B-6.2. The tube is held in place by a 2 mm wide strip of rubber cut from the end of a piece of rubber tubing.

Immerse the thermometer and capillary tube intc the warm oil. Keep the rubber tubing out of the liquid. Stir the oil constantly with the glass stirrer. Continue to heat the melting bath with the burner. The temperatures at which the sample starts to liquefy and at which it is completely liquid are recorded as the melting-point range. The range for most pure compounds does not usually exceed 1°C.

Do three trials of each sample to confirm the melting point range. Record the ranges on the data sheet as well as your observations. Obtain a sample of an unknown and determine which of the three compounds tested it is by doing a melting point determination of the unknown and comparing the data to the others.

DATA

Name of Sample	Trial 1 (°C)	Trial 2 (°C)	Trial 3 (°C)	Observations While Melting
Naphthalene				
Benzoic Acid				
Salicylic Acid				
Unknown				
Analysis of Unknown:				

EXPERIMENT B-7: MELTING BEHAVIOR OF CRYSTALS VS. AMORPHOUS SOLIDS

INTRODUCTION

In the previous two experiments you have examined four different crystals—paradichlorobenzene, naphthalene, benzoic acid, and salicylic acid. These substances have sharp melting point ranges when compared to amorphous solids such as paraffin. Amorphous solids are ones without definite form as opposed to a solid which is crystallized.

MATERIALS

Crystals of paradichlorobenzene; small pieces of paraffin; large petri dishes; hot (80°-90°C) water; two small beakers; 250 ml beaker; thermometer.

INSTRUCTIONS

Set the two beakers in the large petri dish on the lab table. In the meantime, prepare the hot water on your ring stand. Place 2 or 3 crystals of paradichlorobenzene in one of the beakers and a small piece of paraffin in the other beaker.

Pour hot (80°-90°C) water into the petri dish. *Use caution when pouring hot liquids.* Hold down the beakers since they might float and tip over. Observe the sharp melting range of paradichlorobenzene and the broad softening range of paraffin. Time how long it takes for both substances to melt. Record your observations on the data sheet.

Remove the beakers from the hot water and dispose of the hot water. Set the beakers on the cool table top and observe the differences between the two substances as they begin to cool and solidify. This time record how long it takes for the liquids to solidify. Record these observations on your data sheet. Use a magnifying glass to examine both substances more closely. On your data table include a sketch of each of the solidified substances. *Use the proper container for disposing of the crystals and solids. Do not put them down the drain.*

DATA

Melting Behavior Observations	
Paradichlorobenzene	Paraffin
Melting Time:_____	Melting Time:_____

Solidification Behavior Observations	
Paradichlorobenzene	Paraffin
Freezing Time:_____	Freezing Time:_____

Sketchs of Solids Observed Through Magnifying Lens	
Paradichlorobenzene	Paraffin

EXPERIMENT B-8: DISTILLATION

INTRODUCTION

In distillation, a liquid is converted into a vapor by heating it to its boiling point. The vapor is then cooled and the condensed liquid is collected in a suitable container. Any dissolved substances having higher boiling points are left behind as the temperature of pure vapor remains constant during the distillation.

MATERIALS

Distilling flask; thermometer; condenser; two ring stands with ring and clamp; Bunsen burner; test tubes; funnel; rubber tubing; asbestos square; balance; graduated cylinder (50 ml); beaker or gas collecting bottle (any size); boiling stones; tap water; solid potassium permanganate ($KMnO_4$) and sodium chloride (NaCl); ammonium hydroxide solution; phenolphthalein solution.

INSTRUCTIONS

Assemble the apparatus as illustrated in Figure B-8. Before beginning the experiment have your instructor check it to be sure that all parts are properly assembled.

Prepare a solution of tap water, a small crystal of potassium permanganate, and 5.0 grams of sodium chloride. Pour this solution into the distilling flask until it is two-thirds full. *Use a funnel to insure that the solution will not go down the side arm instead of into the flask.* Then add 5 or 6 boiling stones to the solution. Begin to heat the water in the flask. Boil the solution and discard the first 10 ml of distillate (the liquid that condenses). Continue to boil steadily but be sure not to boil over any liquid by heating too rapidly. What is the temperature of the hot vapor? Record all your observations on your data sheet. Collect about 20 ml of the distillate in a clean beaker. Examine it. What is its color? How does it taste?

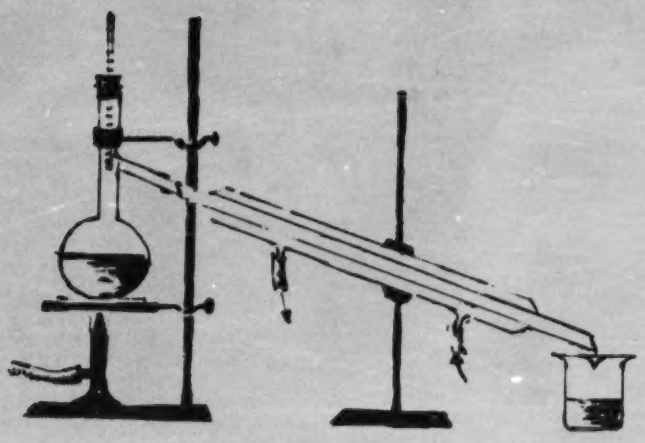


Figure B-8.

Repeat the above procedure after cleaning out the flask. This time fill the flask 2/3 full of water and add to it a drop ammonium hydroxide. To each of six clean test clean test tubes, add 1 drop of phenolphthalein solution, and use these tubes one after another to catch 10 ml of the distillate. Change the tube as soon as 10 ml has been collected. What observations can you make about these six filled test tubes? Record your observations and answer the question pertaining to this second distillation.

DATA

Describe the physical properties of the first distillate.

What was its boiling point?

Did the potassium permanganate vaporize over into the distillate?

Did the sodium chloride vaporize over into the distillate? How you know?

In the second distillation was there evidence that the ammonium hydroxide passed over with the steam? What was the evidence?

Which test tube contained the most ammonia?

Which test tube contained the least ammonia?

Describe what would happen if you were given a mixture of three liquids, each with a different boiling point, and were instructed to distill it.

Can the process of distillation help with the world problem of water shortages and water pollution? How?

EXPERIMENT B-9: DRY DISTILLATION OF WOOD

INTRODUCTION

Destructive distillation is the decomposition that results when an organic substance is heated in the absence of air. Certain industries distill coal or wood. Then the liquid portion of the product is further separated into its components by fractional distillation. The liquid product from coal is known as coal tar and from wood is known as wood tar or pyroligneous acid. When wood is destructively distilled, wood tar and wood gas will be obtained.

MATERIALS

Sawdust; two clamps; ring stand; rubber stoppers (one hole and two hole); glass tubing; two large test tubes; blue litmus paper.

INSTRUCTIONS

Assemble the apparatus as shown in Figure B-9. Place the sawdust in the test tube on the left until it is about half filled. Fill the test tube on the right about one-third full of water. Make sure the jet tube on the right has a small tipped opening.

After everything is assembled, heat the tube with the sawdust uniformly, gently at first. After the gas starts coming over and the tar begins to collect under the water, try to ignite the gas coming out of the tip. What color is the flame?

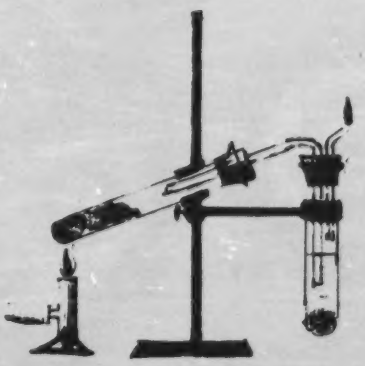


Figure B-9.

After the test tube cools off, examine the residue left in the tube. Examine the wood tar in the tube on the right. Record your observations on the data sheet. Dip a strip of blue litmus paper into the water layer of the right test tube. What happened? Describe the odors of the wood tar. *Be careful not to spill any wood tar on your skin since its penetrating odor will persist despite washing.*

DATA

General Observations During Distillation:
Color of Flame:
Odor of Wood Tar:
Characteristics of Residue Left in Sawdust Tube After Distillation:
Results of Litmus Test:

EXPERIMENT B-10: CHEMICAL CHANGES AND EQUATIONS

INTRODUCTION

There are many ways to detect or observe a chemical change (a change in composition). Some highly precise instruments enable us to measure not only the degree of change in composition, but also the change in energy which takes place when substances react chemically. For many reactions the changes which occur in energy and composition during these reactions are similar. After observing many reactions, we can see a trend in properties and we can develop broad generalizations about the nature of matter. Such generalizations are the heart of chemistry. In this experiment, you will work with three classes of chemical reactions: displacement, decomposition, and synthesis.

MATERIALS

Crucible and cover; wire triangle; forceps; balance; asbestos pad; glass tubing; triangular file; one hole stopper; graduated cylinder; wooden splints; asbestos square; test tubes; wing tip for Bunsen burner; ruler; scissors; magnesium ribbon; limewater; mossy zinc; 6M HCl (hydrochloric acid); graduated cylinder; zinc dust; flowers of sulfur (or sublimed sulfur); solid copper carbonate or magnesium carbonate.

INSTRUCTIONS

Obtain a piece of magnesium (Mg) about 20 cm long and roll it into a loose ball. Place the magnesium in a crucible and cover it. Weigh the crucible, cover, and its contents carefully to the nearest 0.01 gram. Record the weight on the data sheet on line 2. Place the crucible in a triangle, and then put the triangle on a ringstand and ring. Begin heating, slowly at first. For the best results, it is necessary that the magnesium burn *very slowly*, and that the finely divided, white magnesium oxide smoke be kept from escaping. This can be accomplished by heating the bottom of the crucible and lifting the cover only momentarily to allow a fresh supply of air to enter the combustion chamber. Begin by holding the crucible cover with a pair of tongs while you heat the bottom of the crucible until the magnesium ignites. At this moment, place the cover on the crucible. After a short interval lift the cover and allow enough air to enter the crucible to again ignite the magnesium. Immediately replace the cover. Repeat this procedure until the magnesium no longer ignites when you raise the cover. At this point adjust the cover so that most of the crucible opening is covered but there is enough gap to allow a steady flow of air into the crucible. Heat with a hot flame for a few minutes. *Allow to cool.* Remove the crucible from the triangle with forceps, weigh it carefully, and record your data on line 1 of the data sheet. Then empty the contents onto a piece of paper. Examine the contents for a change in composition. Compare the new weight with the old weight. If a reaction did occur, with what did the magnesium react?

From the glass tubing supply, use a triangular file to cut a piece of glass tubing about 15 cm long. Fire polish the ends and bend the glass tubing into a 90° angle. Obtain two small spatulas full of either $CuCO_3$ (copper carbonate) or $MgCO_3$ (magnesium carbonate) and place the solid in a large dry test tube. Insert the glass tubing into a one-hole rubber stopper after lubricating the tubing well. Then insert the stopper into the test tube containing the carbonate. Pour about 5 ml of limewater $Ca(OH)_2$ into a small test tube. Place the end of the right angle glass tube into the limewater solution. (See Figure B-10.1)

Heat the tube containing the carbonate while holding the end of the glass tube in the limewater solution. Remove the tube from the limewater *before* removing the flame from the tube containing the carbonate. A cloudy appearance in the limewater indicates the presence of carbon dioxide, CO_2. Make all the observations you can and record them on the data sheet. Let the test tube cool and empty the contents of both tubes.

After cleaning out the apparatus used above, place a few small pieces of mossy zinc in the test tube on the left and add 5 ml of 6 M HCl (hydrochloric acid). Quickly insert the rubber stopper containing the glass delivery tube. A reaction should be occurring and a gas should escape from the tubing. With the glass tubing turned upwards, collect some of the gas being liberated. (See Figure B-10.2). Collect the gas by displacement of air by inverting another test tube over the up-turned gas delivery tube. Remove the tube from the glass tubing but still keep the test tube inverted. Bring a burning splint near its mouth. A "pop" indicates the presence of hydrogen gas.

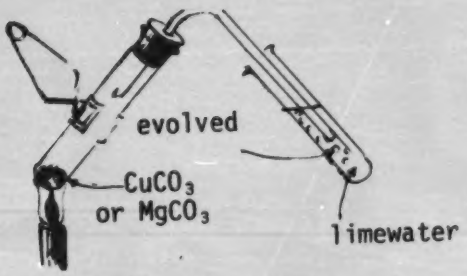


Figure B-10.1.

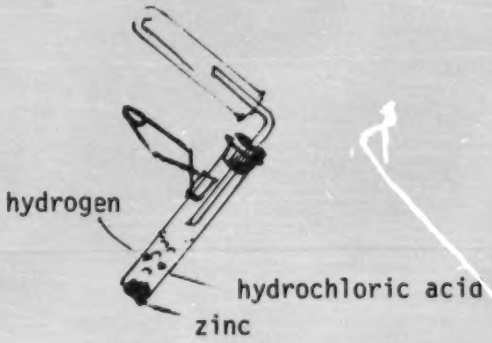


Figure B-10.2.

Weigh out 0.2 gram of zinc dust and 0.1 gram of sulfur. Mix thoroughly but be careful not to grind or crush the materials. Place the mixture on an asbestos square and ignite with a Bunsen burner. *Caution: Once the reaction begins it will be very rapid. Step back from it.*

DATA

Magnesium Reaction	
1. Weight of crucible with magnesium after heating _____ g	
2. Weight of crucible with magnesium before heating _____ g	
3. (Weight after heating) - (Weight before heating) = _____ g	
Properties Before Heating	Properties After Heating
Equation: Magnesium + _____ → Magnesium oxide	

Copper Carbonate Reaction
Observations Upon Heating:
Equations: Copper carbonate $\xrightarrow{heat}$ Copper oxide + _____↑(gas) _____(gas) + Calcium hydroxide → Calcium carbonate + water (limewater)

Zinc and Acid Reaction
Observations Upon Addition of HCl to Zinc:
Equation: Zinc + Hydrochloric acid (HCl) → _____↑(gas) + Zinc chloride

Zinc and Sulfur Reaction
Observations When Heated:
Equation: Zinc + Sulfur → _____

EXPERIMENT B-11: CHEMICAL CHANGE
REDUCTION OF LEAD

INTRODUCTION

The processes of oxidation and reduction are chemical changes. It is possible to study some samples of these changes by investigating several processes in the metallurgy of lead which can be done on a laboratory scale by the proper treatment of a lead compound. The Figure B-11 shows how to direct either the reducing flame or the oxidizing flame down on the compound to be heated.

MATERIALS

Mouth blowpipe; charcoal block; beaker; lead oxide (PbO); powdered charcoal; lead sulfide (PbS); lead carbonate ($PbCO_3$); acetic acid; zinc strips; crucible.

INSTRUCTIONS

Before using a blowpipe, sterilize its mouthpiece by heating it in the burner flame for a few moments. Then cool it under cold water.

Make a depression in a charcoal block and fill it with a thick paste made by mixing about one gram of lead oxide (PbO) and powdered charcoal with a few drops of water. By blowing through the blowpipe, direct the reducing (inner) flame down on the lead oxide-carbon mixture until you observe a change. Since it may take quite a while for a change to occur, you may want to clamp the Bunsen burner in place as Figure B-11 shows. Record your observations.

Clean the depression in the charcoal block and enlarge it if necessary. Fill the depression with a gram of powdered lead sulfide (PbS). Direct the oxidizing (outer) flame down on the lead oxide-carbon mixture for two or three minutes. Observe the results and record them on the data sheet.

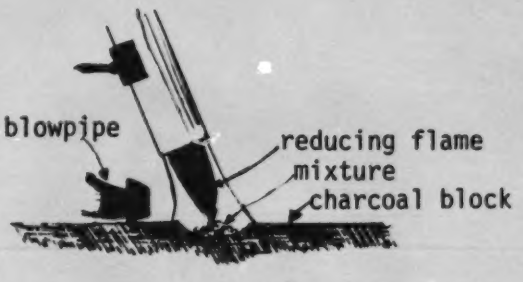


Figure B-11.

In a used crucible heat strongly for a few minutes about one gram of lead carbonate ($PbCO_3$). After the product has cooled, note its color. Transfer the contents of the crucible to a beaker and dissolve it in just enough acetic acid. What compound of lead is formed? After observing the solution, put a zinc strip into the liquid. Let it set for a few minutes. Observe what happens and record all of your observations.

DATA

Lead Oxide Treatment
Observations:
Equations: Lead (II) oxide + Carbon → _____ + _____↑ PbO + C → _____ + _____↑
Is this reaction spontaneous or does it have to be forced?

Lead Sulfide Treatment
Observations:
Equations: Lead (II) Sulfide + Oxygen → _____ + _____↑ PbS + O_2 → _____ + _____↑

Lead Carbonate Treatment
What change in color do you note after heating?
Equations: Lead (II) Carbonate $\xrightarrow{heat}$ _____ + _____↑ $PbCO_3$ $\xrightarrow{heat}$ _____ + _____↑
What did you observe when zinc was added to the lead solution?

EXPERIMENT B-12: CHEMICAL CHANGE
CORROSION OF IRON

INTRODUCTION

Each year the United States alone loses an estimated six billion dollars as a result of the corrosion of steel. Corrosion is a general term applied to the process in which uncombined metals are converted to oxides or other compounds. This causes the gradual deterioration of metals. In this experiment we shall investigate some of the factors involved in corrosion.

MATERIALS

Five new nails; 5 test tubes; red and blue litmus paper; 0.1 M potassium ferricyanide; solutions of Group A, B, or C; distilled water; 0.1 M ferrous sulfate ($FeSO_4$).

INSTRUCTIONS

Either with a china marking pencil or some masking tape, label five test tubes according to the group (A, B, or C) which you have been assigned to study. Place a clean, bright, non-galvanized nail in each of the five test tubes. Slide each nail carefully down the side to avoid breaking the bottom of the test tube. Partially fill each of the test tubes with the appropriate solutions until the nail is just covered. All the solutions are the same concentration. Use red and blue litmus paper to determine whether the solutions in your group are acid (blue turns red) or basic (red turns blue).

Group A	Group B	Group C
1. NaOH	KOH	Na_3PO_4
2. $Na_2Cr_2O_7$	Na_2CO_3	$Na_2C_2O_4$
3. NaCl	KNO_3	KSCN
4. HCl	HNO_3	H_2SO_4
5. Distilled H_2O	Distilled H_2O	Distilled H_2O

Allow the nails to stand overnight in the solutions. Observe and record any changes that have taken place by removing the nails from the solutions, placing them on some toweling, and examining them with a hand lens. To each of the test tubes add one or two drops of 0.1 M potassium ferricyanide, $K_3Fe(CN)_6$. This is the test for the presence of iron in the solution which might have dissolved out of the nails as a result of corrosion. As a control and for comparison, add one drop of 0.1 M potassium ferricyanide solution to about 1 ml of ferrous (Iron II) sulfate solution. Compare this result to that obtained when the potassium ferricyanide was added to the various solutions in which the nails had been setting overnight. Record all of your observations.

DATA

Formulas Group ____	Acid or Base	Evidence of Rust	Other Changes	Result of adding $K_3Fe(CN)_6$	Appearance when Dried
1.					
2.					
3.					
4.					
5. Distilled H_2O					

EXPERIMENT B-13: ALLOTROPIC FORMS OF SULFUR

INTRODUCTION

When atoms or molecules form crystals, the environmental conditions are very important factors and actually determine the form of the resulting crystalline structure. For example, when carbon atoms are slowly forced together under very high pressure and temperature, diamonds are formed. Under other conditions, graphite or coal (which is mostly carbon) is formed. Different crystalline forms of the same substance are termed allotropic forms (diamond and graphite). The factor which most influences the arrangement of the atoms or molecules in a crystal is the bonding characteristics of the atoms.

In this experiment, you will change the conditions under which crystals of the pure element, sulfur, form. As you change the conditions, you will examine the structure or arrangement of the crystals to see if the structure changes.

MATERIALS

Roll sulfur; carbon disulfide (CS_2); test tubes; watch glass; filter paper; 250 ml beaker; magnifying hand lenses; test tube holder; graduated cylinder; corks.

INSTRUCTIONS

Obtain about 5 ml of carbon disulfide, CS_2, in a test tube. *Caution: CS_2 is very flammable and its vapors are poisonous. Check to be sure that there is no Bunsen burner lighted near your laboratory station.* Add a small piece of sulfur, cork the test tube, and shake until most of the sulfur has dissolved. Decant some of the clear liquid into a watch glass and place the glass under a hood or near an open window. Allow the watch glass to stand until all of the CS_2 has evaporated.

Select an old test tube or one supplied by your teacher and fill it about one-third full of sulfur. Fold a piece of filter paper into the conical shape used when filtering with a funnel. Heat the test tube until all of the solid sulfur has melted. Pour the liquid rapidly into the filter paper. As the sulfur begins to solidify, break the paper open *carefully*. Examine the sulfur with a hand lens. Record your observations.

Place more sulfur into the same test tube. Use about the same amount of sulfur as before. Heat this sample of sulfur until it just begins to boil. Immediately pour the hot sulfur into a 250 ml beaker filled about three-fourths full of water.

Examine the different samples for crystals with a microscope or magnifying lens. Also examine other crystals which your instructor may have avalable.

DATA

Observations of the three forms of sulfur crystals:		
Evaporated from CS_2	Poured hot on filter paper	Poured into cold H_2O

Drawings of the three forms of sulfur crystals observed:		
Evaporated from CS_2	Poured hot on filter paper	Poured into cold H_2O

What other environmental conditions besides temperature can be changed to produce allotropes?

EXPERIMENT B-14: THE RATE OF A REACTION

INTRODUCTION

The *rate of a reaction* is defined as the quantity of reactant consumed or product formed during some stated time interval at a fixed temperature or temperature range. In this experiment, you will investigate the effects of various conditions on the rate of consumption of magnesium in the reaction between magnesium and hydrochloric acid.

MATERIALS

Magnesium ribbon; 0.5 M hydrochloric acid (HCl); 1 M hydrochloric acid; ice; test tubes; 250 ml beakers; graduate cylinders; thermometer; timer; ruler; scissors; burner; matches; stirring rod.

INSTRUCTIONS

EFFECT OF TEMPERATURE

Add 10 ml of 1 M hydrochloric acid to a test tube. Place the tube in a 250 ml beaker half-filled with tap water. After 2-3 minutes, record the temperature of the acid. Remove the thermometer and place a 2 cm strip of magnesium ribbon into the test tube. Be sure the metal remains below the surface of the acid throughout the experiment--use your glass stirring rod to hold it down if necessary. Record the time required for the magnesium to be consumed. Record the final temperature of the solution in the test tube. Repeat the experiment at temperatures near 0°C, 50°C, and 100°C using an ice bath and hot water bath. Since you are recording the temperature range of the acid solution, it is unnecessary to maintain the initial temperature of the solution.

EFFECT OF CONCENTRATION

Pour 16 ml of 1 M hydrochloric acid into a test tube. Place the tube in a 250 ml beaker half-filled with tap water. Insert a strip of magnesium ribbon into the test tube. Be sure the metal remains below the surface of the acid throughout the experiment. Record the time required for the magnesium to be consumed. Prepare the following solutions of 1 M hydrochloric acid and water. Mix thoroughly and repeat the experiment with each solution: 12 ml of acid and 4 ml of water, 10 ml of acid and 6 ml of water, and 8 ml of acid and 8 ml of water.

EFFECT OF SURFACE AREA

Place a test tube containing 10 ml of 0.5 M hydrochloric acid in a 250 ml beaker half-filled with tap water. Roll one of the magnesium strips into a tight ball and drop it into the test tube. Be certain that the metal remains below the surface of the acid throughout the experiment. Record the time required for the magnesium to be consumed. Repeat the experiment, using a flat strip of magnesium. Repeat once more with another strip of magnesium cut into as many slivers as possible.

DATA

Effect of Temperature:		
Tube	Temperature Range (°C)	Reaction time (sec)
1		
2		
3		
4		

Effect of Concentration:			
Tube	ml of Acid	ml of Water	Reaction time (sec)
1			
2			
3			
4			

Effect of Surface Area:		
Tube	Shape of Magnesium	Reaction time (sec)
1		
2		
3		

Equation:
Magnesium + Hydrochloric Acid → _____ + _____ ↑
_____ + _____ → _____ + _____ ↑
(Don't forget to *balance* the equation.)

EXPERIMENT B-15: A STUDY OF REACTION RATES

INTRODUCTION

To what extent do changes in the concentration of the reacting substances affect the rate of a chemical reaction? In this experiment you will investigate the role of such factors as concentration and temperature by performing some experiments with an interesting reaction called the "clock reaction." You will appreciate the significance of this name after you have completed the experiment. In order to determine the role of each factor independently, you will vary the concentration of one of the reactants in the first part and vary the temperature in the second part. In each case you will try to keep other possible variables constant.

MATERIALS

Solution A; Solution B; distilled water; ice; test tubes; 250 ml beaker; graduated cylinder; 2 thermometers; timer; burner; ring stand and ring; wire gauze; corks.

INSTRUCTIONS

Use a clean graduated cylinder to measure 10 ml of Solution A and pour it into a clean test tube. Rinse the graduated cylinder thoroughly and in a similar manner place 10 ml of Solution B into another test tube. Pour Solution B into Solution A quickly, cork, shake and record the time to the nearest second that it takes for a reaction to begin. Do a second trial of this mixing to test your accuracy. Prepare five different concentrations of Solution A by diluting Solution A as follows:

(1) 9 ml of A with 1 ml of distilled water
(2) 8 ml of A with 2 ml of distilled water
(3) 7 ml of A with 3 ml of distilled water
(4) 6 ml of A with 4 ml of distilled water
(5) 5 ml of A with 5 ml of distilled water.

Note that the volume is always 10 ml. Mix each of the diluted solutions well. Now repeat the mixing by adding 10 ml of Solution B to each of the solutions made above. Record the time it takes for each reaction on your data sheet.

In order to investigate the effect of changes in temperature, you will first determine the time of this reaction at room temperature, then at a temperature within twenty degrees below room temperature, and lastly at a temperature within twenty degrees above room temperature. Put 10 ml of Solution A into one test tube and 10 ml of Solution B into another. This solution should be placed in room temperature water in a beaker. Once they have reached the room temperature, quickly pour Solution B in Solution A, cork, and quickly shake, recording how many seconds it took for a reaction to appear. Prepare a water bath at the new cooler temperature and repeat the

above procedure once the tubes and solutions have reached the new cooler temperature. Record your results. Prepare a water bath at the new hotter temperature and repeat once again. Record all of your observations on the data sheet.

Prepare graphs of your data. See Figure B-15.1 and Figure B-15.2 for instructions on labeling your axes.

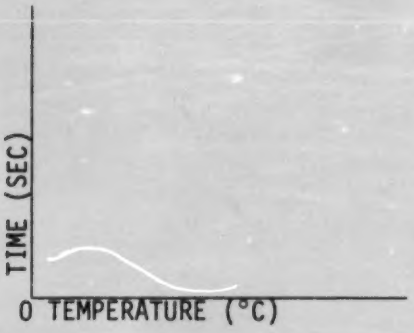


Figure B-15.1.

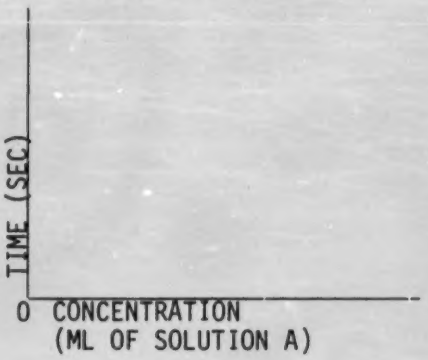


Figure B-15.2.

DATA

ml of Solution A	ml of Water	ml of Solution B	Time for Reaction	
			Trial 1	Trial 2
10	0	10		
9	1	10		
8	2	10		
7	3	10		
6	4	10		
5	5	10		

ml of Solution A	ml of Solution B	Temp. of Solutions	Time for Reaction	
			Trial 1	Trial 2

EXPERIMENT B-16: CATALYSTS

INTRODUCTION

A catalyst is a substance that changes the rate of a chemical reaction and that can be recovered unchanged at the end of the reaction. Catalysts may be classified as: surface or contact catalysts, which furnish a surface at which the reactants concentrate and react; and "carrier" catalysts, which form reactive intermediates that decompose into products and regenerate the catalyst. Sometimes the catalyst is "generated" by one of the reactants, and " self" or autocatalysis is noted.

Most chemical reactions that occur in plant and animal tissue are stimulated or retarded by chemicals called catalysts. Chlorophyll, the green coloring material in leaves of plants, is one of the best-known plant catalysts. Many other chemical reactions are also affected by catalysts. In fact, we now know that most reactions can be catalyzed. In this experiment we will investigate the effect of a variety of chemicals as catalysts.

MATERIALS

Powdered manganese dioxide (MnO_2); copper (II) oxide (CuO); 3% hydrogen peroxide (H_2O_2); calcium oxide (CaO); cubes of sugar; iron oxide (Fe_2O_3); chromium oxide (Cr_2O_3); sodium bicarbonate ($NaHCO_3$); potassium chlorate ($KClO_3$); 18 M sulfuric acid (H_2SO_4); 0.15 M oxalic acid ($H_2C_2O_4$); 0.001 M potassium permanganate ($KMnO_4$) solution; crystals of manganese (II) chloride ($MnCl_2 \cdot 4H_2O$); tongs; test tubes; test tube holder; wooden splints; graduated cylinder; burner; matches; timer.

INSTRUCTIONS

Fill the rounded end of a Pyrex glass test tube with potassium chlorate ($KClO_3$) and mix with it about one half as much iron oxide (Fe_2O_3). Place the mixture in the pyrex test tube and heat it *gently*. With a glowing splint, test the gases in the test tube for the presence of oxygen. If the splint flares into a flame oxygen is present. Now repeat this experiment with the following oxides, trying each one individually with potassium chlorate: manganese dioxide (MnO_2); calcium oxide (CaO); copper (II) oxide (CuO); and chromium oxide (Cr_2O_3). Record your observations on the data sheet.

Pour about 3 ml of a 3% solution of hydrogen peroxide, H_2O_2, into a test tube and add about 1.5 grams of manganese dioxide (MnO_2) to it. Mix these by rotating the test tube. Test for the evolution of oxygen with a glowing splint. Repeat this procedure using a fresh sample of hydrogen peroxide and sample of the other oxides. Record your observations.

Grasp a cube of sugar with a pair of crucible tongs. Hold it in the edge of a flame from a burning wooden splint, and note whether it can be ignited readily. Turn the cube around in the tongs to expose an unheated corner; on this corner sprinkle some powdered sodium bicarbonate. Now hold this corner of the cube in the edge of the flame from a burning wooden splint, and attempt to ignite the sugar. Record your observations.

To a clean flask add 25 ml of 0.15 M oxalic acid solution. *Caution: Add 1 ml of concentrated 18 M sulfuric acid solution to the flask very carefully!!* Then add 10 ml of 0.001 M potassium permanganate ($KMnO_4$) solution, swirl the contents of the flask, and note the time required for a noticeable color change. Also record the time for the pink color to disappear completely. Repeat this experiment, but this time, after the permanganate solution is added, quickly add a very small crystal of hydrated manganese (II) chloride ($MnCl_2 \cdot 4H_2O$). Swirl the contents of the flask and note the time required for the pink color to disappear.

DATA

Potassium Chlorate Plus Catalysts		
Name of Catalyst	Observations	Oxygen present
Iron Oxide		
Manganese Dioxide		
Calcium Oxide		
Copper (II) Oxide		
Chromium Oxide		

Hydrogen Peroxide plus Catalysts		
Name of Catalyst	Observations	Oxygen present
Manganese Dioxide		
Chromium Oxide		
Calcium Oxide		
Copper (II) Oxide		
Iron Oxide		

Ignition of Sugar	
Results without catalyst:	Results with catalyst:

Autocatalysis of Oxalic Acid - Potassium Permanganate Reaction	
Time required for significant color change (no manganese (II) ion added)	_____min.
Time required for color change (manganese (II) ion added)	_____min.

EXPERIMENT B-17: AN EXPANDED STUDY OF REACTION RATES

INTRODUCTION

In this experiment we shall continue our study of the factors which influence the rate of a reaction by investigating the rate of decomposition of a commercial bleaching solution. Since the rate of this reaction is extremely slow under normal conditions, we shall use a solid catalyst, an oxide of cobalt, to catalyze each of the reactions. The catalysis will be prepared right in the reaction vessel; we wish to start the decomposition by reacting some of the bleach with a few milliliters of a solution of cobalt nitrate. The rate of decomposition of the bleach will be observed by following the volume of water displaced by the oxygen formed during time intervals. The effects of changing the temperature and the concentration will be studied.

MATERIALS

Two 250 ml Erlenmeyer flasks; screw or pinch clamp; small test tube; rubber tubing; glass tubing; one and two hole stoppers; thermometer; ring stand and clamp; graduated cylinder; timer; commercial bleach solution; 0.17 M ferric nitrate solution; methyl orange indicator; 0.17 M cobaltous nitrate solution; ice.

INSTRUCTIONS

Assemble the apparatus as illustrated in Figure B-17. Ask your instructor to check it before beginning the experiment.

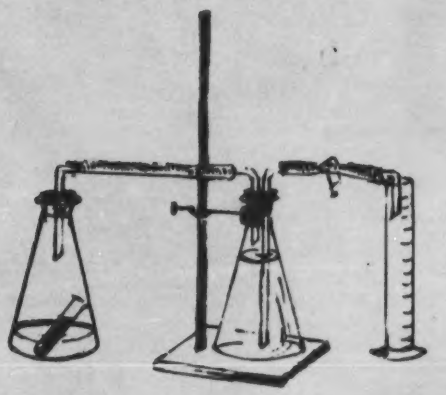


Figure B-17.

Measure carefully 15 ml of the bleach solution into a graduated cylinder, determine its temperature, then pour it into the reaction flask, the first Erlenmeyer flask on the left. Place 3 ml of the cobalt nitrate solution in the small test tube and insert it carefully into the same Erlenmeyer flask *without spilling any of the contents*. Fill the second (middle) Erlenmeyer flask full of water. Add a few drops of methyl orange indicator to the water in order to make it easier to read the water level in the cylinder when it begins to come over.

Open the pinch clamp and prime the delivery tube leading to the graduated cylinder by blowing into the short glass elbow inserted in the reaction flask. Close the pinch clamp while the delivery tube is full of water.

Secure all connections and check that the rubber stoppers fit tightly in the flasks. Note the time as you quickly tip the reaction flask slightly to pour the cobalt nitrate solution out of the test tube into the bleach. Remove the pinch clamp. Hold the erect flask at its neck and move it constantly with a gently swinging motion to dislodge the gas bubbles from the solid catalyst in a uniform manner. This motion must be gentle and uniform in all trials. Record the volume of water in the graduated cylinder every 30 seconds until 50 ml has been collected.

Rinse out the reaction flask and repeat the procedure as directed except have the temperature of the solutions in the reaction flask ten degrees centigrade above room temperature by warming the reaction flask in a lukewarm water bath.

Repeat the procedure with the solutions in the reaction flask at ten degrees below room temperature. Cool the reaction flask and the solutions in a cold water bath. Obtain some ice if necessary.

Repeat the procedure at room temperature as in the above except add 18 ml of water to the bleach solution in the reaction flask in order to obtain a two-fold dilution in the final volume of bleach.

Repeat the procedure at room temperature but use 3 ml of 0.17 M ferric nitrate in place of the cobalt nitrate solution.

Repeat the procedure but this time try a combination of cobalt and ferric nitrate (1 ½ ml of ferric nitrate and 1 ½ ml of cobalt nitrate mixed). This would precipitate mixed oxides as possible catalysts. Record all data on your data sheet.

Hint: Keep your apparatus together and check with your instructor to find out whether you will be doing the next experiment, because only a slight modification of your apparatus will save you a great deal of time when doing *The Effect of the Amount of a Catalyst*, Experiment B-18.

DATA

Room Temp.		Above Rm. Temp.	Below Rm. Temp.	Dilution	Ferric Nitrate	Mixed Catalysts
Time	Volume	Volume	Volume	Volume	Volume	Volume
0 sec						
30 sec						
1 min						
1½ min						
2 min						
2½ min						
3 min						
3½ min						
4 min						
4½ min						
5 min						
5½ min						
6 min						
6½ min						
7 min						
etc.						

EXPERIMENT B-18: THE EFFECT OF THE AMOUNT OF A CATALYST

INTRODUCTION

In this experiment you are faced with the problem of determining how the amount of a catalyst present may affect the rate of a chemical reaction. Let us imagine that a scientist faced this problem and that he designed what he considered to be a reasonable experiment. The reaction he chose to study was that of the decomposition of hydrogen peroxide. If we can assume that the volume of oxygen gas given off in a specified interval will indicate a measure of the rate of decomposition, then this knowledge presents an easy method of measuring the rate. One of the substances that catalyzes this reaction is manganese dioxide. Thus we have the information necessary to devise a procedure to carry out the experiment.

MATERIALS

The students should keep the apparatus intact from the previous experiment, *An Expanded Study of Reaction Rates*. In addition, the following items are needed: hydrogen peroxide solution (3%); powdered manganese dioxide.

INSTRUCTIONS

Remove the small test tube from the reaction flask in Figure B-17. Leave all other items intact from the previous experiment. See the previous experiment for the details on how to be sure that the system is secure and ready for use. Have your instructor check your system. Add the manganese dioxide (2.0 grams) to the dry reaction flask. After it is added, pour 10 ml of a 3% hydrogen peroxide solution quickly into the flask, stopper it immediately and prepare to record data. Open the pinch clamp and begin to measure and record the volume of water collected in the cylinder every 15 seconds.

Repeat the above procedure, but this time use 1.5 grams of catalyst instead of 2.0 grams. Record all the volume readings on the data sheet.

Finally, repeat the entire procedure again but use only 1.0 grams of catalyst.

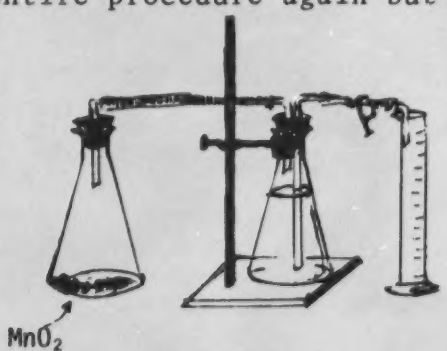


Figure B-18.

DATA

2.0 gram of catalyst		1.5 gram of catalyst	1.0 gram of catalyst
Time	Volume	Volume	Volume
0 sec			
15 sec			
30 sec			
45 sec			
1 min			
$1\frac{1}{4}$ min			
$1\frac{1}{2}$ min			
$1\frac{3}{4}$ min			
2 min			
$2\frac{1}{4}$ min			
$2\frac{1}{2}$ min			
$2\frac{3}{4}$ min			
3 min			
$3\frac{1}{4}$ min			
$3\frac{1}{2}$ min			
$3\frac{3}{4}$ min			
4 min			
etc.			

EXPERIMENT B-19A: CONVERSION OF STARCH TO GLUCOSE ENZYMES AS CATALYSTS

INTRODUCTION

Starch is made of large molecules that are called polymers of glucose, $C_6H_{12}O_6$ (see Figure B-19). Starch can be converted to glucose by adding water to starch which will be absorbed in its connective joints. This reaction is called hydrolysis of starch. The hydrolysis reaction is speeded up to very rapid rates by means of a catalyst called an enzyme. Such an enzyme, called ptyalin, is present in the saliva. By use of this enzyme, starch foods are rapidly hydrolyzed to glucose in the mouth and stomach. In this experiment you will learn of a test for starch and for glucose; then with your own saliva, you will catalyse the hydrolysis to glucose of a sample of starch.

MATERIALS

Fehling's solution A and B; 20% acetaldehyde solution; glucose solution; 1% starch solution; dilute iodine solution; ice; distilled water; paraffin; thermometer; funnel; filter paper; beakers; droppers; test tubes; graduated cylinders; thermometer (°C); timer; stirring rod; test tube holder; burner; matches.

INSTRUCTIONS

Test for Starch: A very sensitive test for starch is the deep blue color it forms when it comes in contact with a very dilute iodine solution. Add one drop of iodine solution to 5 drops of a starch solution. Observe the results and record them.

Test for Glucose: The usual test for glucose depends upon the reducing action of the formaldehyde group on the glucose molecule. This is often done with a solution called Fehling's solution which contains copper ions that are reduced to cuprous (copper (I)) and precipitates as red Cu_2O. The appearance of this red precipitate is evidence of the reducing formaldehyde group, and this is the evidence of glucose.

Mix 1 ml each of Fehling's solutions A and B in a test tube. Add one drop of 20% acetaldehyde solution, a drop at a time, warming the mixture after each addition, until the blue color is gone from the solution.

Mix 5 ml of Fehling's solution A with 5 ml of Fehling's solution B in a test tube and add a few drops of a glucose solution. Place the tube in boiling water and observe the color changes taking place.

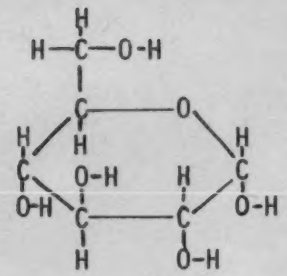


Figure B-19.

If the blue color is not all discharged, add more glucose solution and repeat the heating.

Repeat the above paragraph but use 5 ml of 1% starch solution instead of the glucose.

Test for Conversion of Starch to Glucose Catalyzed by Ptyalin: Wash out your mouth with distilled water. Chew a small piece of paraffin to stimulate the flow of saliva, and place the saliva into a funnel fitted with a piece of filter paper suspended over a small beaker. Use the filtered saliva for the experiments which follow.

Place 10 ml of 1% solution of soluble starch in a 100 ml beaker. Place the beaker and its contents in a large beaker or trough of water heated to about 40°C. Add 10 drops of filtered saliva to the starch solution. At one-half to one minute intervals, remove a drop of the solution by means of a stirring rod, touch it to one of several drops of a very dilute iodine solution placed on a white porcelain surface. When the mixture no longer gives a color with iodine, test 5 ml of it for the presence of a reducing sugar with Fehling's solution. Also, as a control, test a little of starch solution with Fehling's solution. The progressively decreasing intensity of the iodine color in the starch test indicates that the starch is being digested and that other products are being formed which do not give the blue color with iodine. The Fehling's solution test confirms the presence of glucose.

Repeat the above paragraphs using three samples of the starch-saliva mixture--one of which is kept in ice water, one at room temperature, and one at 40°C. Note carefully the time of disappearance of the iodine-starch color. After the tube kept at room temperature no longer gives any iodine color, transfer the first tube from the ice water to the 40°C water bath. Continue to test it for iodine color.

DATA

Test for Starch	
Observations:	
Test for Glucose:	
Observations:	
(a) aldehyde	
(b) glucose	
(c) starch	
Test for Conversion of Starch to Glucose Catalyzed by Ptyalin:	
Observations:	
Time Data:	Ptyalin at Cold Temperature
	(a) Temperature _____°C
	(b) Time needed _____
	Ptyalin at Room Temperature
	(a) Temperature _____°C
	(b) Time needed _____
	Ptyalin at 40°C
	Time needed _____

EXPERIMENT C-1: DIFFUSION OF GASES

INTRODUCTION

According to the kinetic theory, the atoms or molecules of gases travel randomly in straight lines between collisions. But, due to collisions, or near collisions with other atoms or moleucles, the particles follow a zig-zag path. This action increases the time required for particles of a gas to travel from one point to another. The movement of the particles of one material through the particles of some other materials (a medium) is called diffusion. The rates of diffusion for different gases in the same gas medium are different. In this experiment, you will determine exactly the relative rates of diffusion of the two gases, hydrogen chloride and ammonia.

MATERIALS

Glass tubing; burner; matches; cotton; timer; china marker; ring stands; burette clamps; ruler; forceps or tongs; concentrated hydrochloric acid (HCl); concentrated ammonium hydroxide (NH_3) solution; distilled water; acetone.

INSTRUCTIONS

Obtain one length of dry glass tubing. Fire polish both ends. *Caution: It requires a high temperature to soften glass sufficiently for melting and glass cools to a safe handling temperature very slowly.*

A small wad of cotton should be rolled into a long plug of the proper size to fit into the fire polished ends of the tube. Then assemble the apparatus as shown in Figure C-1. Holding the cotton with tweezers, forceps, or tongs, dip one of the cotton plugs into the concentrated HCl solution. *Do not take any of the HCl or NH_3 to your station! Be careful—fumes of both solutions are very irritating if inhaled.* Dip the other cotton into the concentrated ammonium hydroxide solution. Insert the wads into opposite ends of the tube *at exactly the same time.* At the point where the two gases meet, a reaction will occur. Mark the point where the reaction begins with a wax pencil. It might take from two to five minutes for a reaction to start. You should be looking for a faint white ring to form. The cloud will migrate down the tube, so every attempt should be made to detect the white cloud as it is initially being formed.

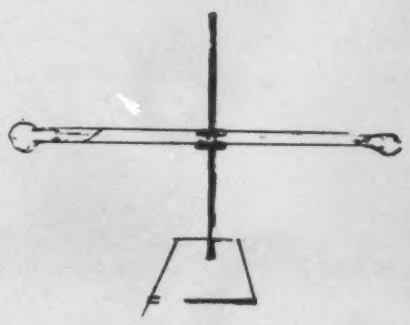


Figure C-1.

After you have observed the reaction and marked the tube where it first occurred, also mark the ends of the tube where the end of the cotton wad is located. Then remove the wads and rinse them under a faucet and dispose of them. With a ruler measure carefully the distance from each end to the mark where the white cloud was first seen. Be certain that you know which end had the hydrochloric acid and which had the ammonium hydroxide. Record your data and perform the necessary calculation.

Rinse the tube with water, then with acetone, and return it to the supply.

DATA

Observations of Reaction:
Time for reaction to occur: _____ min.
Distance traveled by NH_3 molecules: _____ min.
Distance traveled by HCl molecules: _____ min.
$\frac{\text{Distance traveled by } NH_3}{\text{Distance traveled by HCl}} =$ _____

EXPERIMENT C-2: MODELS OF SOME MOLECULES

INTRODUCTION

Chemists find the use of models helpful in studying molecular structure. Many types of molecular models are available. Some models stress the relative sizes of atoms; other models stress bond angles and bond distances. At best, all models are approximations and guides and must be regarded as such.

MATERIALS

A set of molecular models; ruler; protractor.

INSTRUCTIONS

Open your set of models and examine the contents. Record the number of differently colored spheres in the data table. Each hole in a sphere represents a single bond. Count the number of holes present in the different spheres. On the basis of the number of holes, specific spheres have been assigned to represent different elements. Fill in the data table accordingly.

Construct a model of the methane molecule (CH_4). Remove two outer balls and pegs and place the remainder of the model on a clean piece of paper so that it rests on the center sphere and the two outer spheres. Using a sharp pencil, trace the angle formed by the two pegs connecting the spheres. Remove the model and extend the lines on the paper until they intersect. Measure the angle formed with a protractor.

Connect two CH_3 groups with a peg. Draw the image of C_2H_6 on your data table. Construct a model of the butane (C_4H_{10}) molecule. Measure its length and its width.

Contstruct a model for each of the following compounds:

C_2H_2	Acetylene
CO_2	Carbon Dioxide
H_2O	Water
NH_3	Ammonia
HCl	Hydrogen Chloride

Fill in the data table with the requested information and sketches.

DATA

Color of Sphere	No. of Holes Present	Element Represented
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		

Methane— CH_4
Angle Sketch:
_____° = angle between C and two H's
Ethane— C_2H_6
Sketch:
Butane— C_4H_{10} Length = _____ cm Width = _____ cm

Molecular Formula	Name	Sketches
	Acetylene	
	Carbon Dioxide	
	Water	
	Ammonia	
	Hydrogen Chloride	

EXPERIMENT C-3: THE PREPARATION AND INVESTIGATION OF TWO ISOMERS

INTRODUCTION

Maleic acid and fumaric acid have the same molecular formula: $C_4H_4O_4$. Yet each possesses its own distinctive properties, such as melting point, solubility, density, and stability. Chemists attribute these differences to the geometry, or arrangement in space of the atoms in the molecules. They call these molecules isomers of each other.

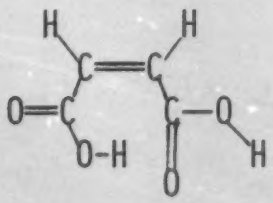
In this experiment you will convert some maleic acid to fumaric acid by heating it in a water solution containing some hydrochloric acid. The hydrochloric acid is not used up by the reaction, but serves merely as a source of H^+ which is the catalyst. You will be able to determine the completeness of the conversion by comparing the weight of the fumaric acid obtained with that of the maleic acid initially used. You will also compare some of the properties of each acid and attempt to explain any differences in terms of their structure.

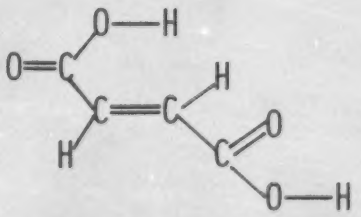
MATERIALS

Solid maleic acid; 12 M hydrochloric acid; magnesium ribbon; solid sodium carbonate; 0.5 M NaOH; phenolphthalein indicator; oil or paraffin; distilled water; buret; burette clamp; ring stand with ring; burner; matches; balance; funnel; filter paper; aspirator (if possible with Buchner funnel for faster filtering); 250 ml Erlenmeyer flask; thermometer; capillary tubes; rubber stopper; graduated cylinder; watch glass; wash bottle; test tubes; beakers; molecular model sets.

INSTRUCTIONS

Construct models of the following molecules from a molecular model set:





Weigh 6.0 grams of maleic acid in a clean, dry beaker. Add 10 ml of distilled water and warm slightly to dissolve the acid. Add 15 ml of 12 M HCl and cover the beaker with a watch glass in the concave position. Place the beaker inside a 250 ml beaker which is about one-third full of water. Heat this water bath to boiling for about 5 minutes or until a solid material forms in the smaller

beaker. Cool the solution to room temperature by placing the smaller beaker in a cold water bath. Filter the mixture. Wash any remaining solid into the filter by rinsing the beaker with small amounts of cold water from a wash bottle. Allow the crystals to drain. Transfer the crystals to a *weighed* watch glass. Dry the crystals by placing the watch glass over an appropriate sized beaker about one-third full of boiling water. When the sample is dry, weigh it on the watch glass. Label the sample "Fumaric Acid."

Compare the solubility of the two acids by placing 0.1 gram of each into separate, properly labeled test tubes. Add 10 ml of distilled water to each, cork and shake. Make a comparison of solubility and record your observations on the data table.

Compare the melting points of the acids using the apparatus shown in Figure C-3. (Look up experiment B-6, *Melting Point*, for details on procedure.) Compare the chemical properties of the two isomers as follows:

Prepare a solution of maleic acid by adding 0.1 gram of maleic acid to 20 ml of distilled water. Divide the solution into two test tubes. To one test tube add a 2 cm strip of magnesium ribbon. Record the results. To the other test tube add a small amount of sodium carbonate. Record the results. Repeat the above tests, using fumaric acid. Record the results and compare them with those obtained with maleic acid.

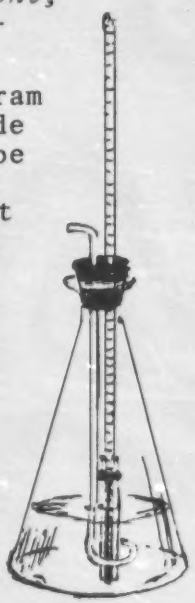


Figure C-3.

Titrate a 0.5 gram sample (dissolved in 25 ml of distilled water) of each acid with a standard sodium hydroxide (0.5 M NaOH) solution using phenolphthalein as the indicator. In order to see a sharp endpoint in this titration of colorless to light pink, place some white paper under the flask which holds the 0.5 gram sample of acid, the 25 ml of water and the 3 drops of phenolphthalein solution. When filling the buret with NaOH, use a small funnel to avoid spilling. Remember to clean the buret thoroughly before using with distilled water and then with a small sample of the NaOH. Record the number of ml of the 0.5 M NaOH necessary to reach the pink endpoint— an endpoint which should stay for at least 30 seconds.

DATA

Preparation:	
Weight of watch glass plus dried fumaric acid = _____ grams	
Weight of watch glass = _____ grams	
Weight of dried fumaric acid = _____ grams	
Solubility:	
Maleic Acid:	Fumaric Acid:
Melting Points:	
Melting point of fumaric acid _____ °C	
Melting point of maleic acid _____ °C	
Reaction with Magnesium:	
Maleic Acid:	Fumaric Acid:
Reaction with Sodium Carbonate:	
Maleic Acid:	Fumaric Acid:
Titration:	
Number of ml of 0.5 M NaOH necessary to neutralize one gram of maleic acid = _____ ml	
Number of ml of 0.5 M NaOH necessary to neutralize one gram of fumaric acid = _____ ml	

EXPERIMENT C-4: MOLECULAR LAYERS

INTRODUCTION

Molecules and atoms are so small that we cannot see them or measure their size except by indirect methods. However, we can get an indication of the order of magnitude of the dimensions of some molecules.

A small quantity of oil placed on the surface of water will spread out to form an exceedingly thin film. The thickness of this film is at least equal to the thickness of one oil molecule. Hence, if we can find the thickness of the layer of the oil we can conclude that the thickness of the oil molecule is equal to, or less than, the thickness of the film.

Oleic acid ($C_{18}H_{34}O_2$) is a good example of easily available material that will form a thin film (see Figure C-4.1 for the structure of oleic acid). One drop of pure oleic acid dropped into a small swimming pool will cover its whole surface. Of course, if the whole surface of the pool is covered, you are not sure that the film could not be thinner. To get the thinnest possible film in a small container, we shall use a drop of a dilute solution of oleic acid in alcohol.

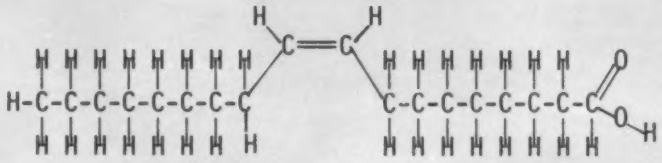


Figure C-4.1.

MATERIALS

Glass tubing; eyedropper; shallow tray or dish; graduated cylinder; ruler; oleic acid solution; alcohol solution; lycopodium powder.

INSTRUCTIONS

Using the glass tubing, make an eyedropper with a very small opening. Now count how many drops of the oleic acid solution will exactly 1 ml in your graduated cylinder.

Fill a large, clean, shallow tray or dish with water (see Figure C-4.2) to a depth of about 1 cm. Dust the surface of the water *lightly* with lycopodium powder. This powder will make the film of oleic acid visible. To be certain that the film will be caused by the oleic acid and not the alcohol, drop one or two drops of alcohol into the tray with an eyedropper. What do you observe?

Figure C-4.2.

Now use the clean eyedropper which you have made according to the instructions above to apply a drop of oleic acid solution. Measure the average diameter of the circular acid film and calculate its area. Do two drops form twice the area that one drop does? How about three drops? What conclusions can you draw?

DATA

Calculations:
_____ drops = 1 ml
Volume = 1 drop = $\frac{1.0}{?}$ ml = _____
Diameter of oil drop = _____ cm
Radius = r = $\frac{\text{diameter}}{2}$ = _____ cm
Area of a circle = A = $\pi \times r^2$ = $\pi \times r \times r$ A = _____ cm^2
Volume = Area × Thickness Thickness = $\frac{\text{Volume}}{\text{Area}}$ = _____ cm
One oleic acid molecule is _____ cm thick!

EXPERIMENT C-5: THE PREPARATION AND STUDY OF CRYSTALS

INTRODUCTION

In crystalline solids, the atoms, molecules, or ions vibrate about fixed positions which are arranged in an orderly fashion. The basic pattern of atoms, ions, or molecules in a crystal which is repeated throughout a crystal is called the space lattice. There are seven different crystal systems and fourteen different types of space lattices. Crystalline solids may be classified in terms of the units that occupy the lattice points and the forces that bind the units together.

MATERIALS

Test tubes; watch glasses; glass slides or glass squares; china markers; microscopes; overhead projector; glass microscope slides; glass stirrer; graduated cylinders; test tube rack; straight pins.

Non-saturated solutions of the following:

(1) Potassium aluminum sulfate
(2) Ammonium aluminum alum sulfate
(3) Magnesium sulfate
(4) Barium chloride
(5) Calcium chloride
(6) Strontium chloride
(7) Sodium potassium tartrate
(8) Sodium chloride
(9) Sucrose
(10) Borax
(11) Disodium phosphate
(12) Copper sulfate
(13) Potassium chromium sulfate
(14) Nickel sulfate
(15) Potassium ferricyanide
(16) Cupric chloride
(17) Sodium sulfite

Saturated solutions of the following:

(1) Calcium chloride
(2) Barium chloride
(3) Strontium chloride
(4) Magnesium sulfate

INSTRUCTIONS

Obtain 1 ml of the non-saturated solutions listed above. Place them in marked test tubes. Mark glass slides or squares with a china marker using a devised code so that you can tell which chemical is on which slide. Place three drops of a solution on the slide. Pour the rest of the solution into a watch glass which is also properly labeled. Allow the slides and watch glasses to dry overnight. When dry use a magnifying glass to examine the crystals in the watch glasses and a microscope to view the crystals on the slides. It is also possible to get a magnification of the crystals on the slides if they are placed on an overhead projector. Observe all the crystals and record your observations.

Place a clean, dry slide on the microscope. Place 1 drop of the saturated barium chloride solution and the magnesium sulfate solution close to each other on the slide but not touching. View the slide under the microscope while you coax the two drops with two straight pins until they touch. Record all of your observations. Allow the drops to evaporate and observe the results.

Repeat the above paragraph with strontium chloride solution and magnesium sulfate.

Also repeat the above procedure with calcium chloride and magnesium sulfate. This is a slow reaction, so be patient. It might take 10-15 minutes. It is worth waiting for.

DATA

Substance	Observation after Evaporation	
	With Microscope	With Hand-Lens
1. Potassium aluminum sulfate		
2. Ammonium aluminum alum sulfate		
3. Magnesium sulfate		
4. Barium chloride		
5. Calcium chloride		
6. Strontium chloride		
7. Sodium potassium tartrate		
8. Sodium chloride		
9. Sucrose		
10. Borax		
11. Disodium phosphate		
12. Copper sulfate		
13. Potassium chromium sulfate		
14. Nickel sulfate		
15. Potassium ferricyanide		
16. Cupric chloride		
17. Sodium sulfite		

Mixtures	Observations
Barium chloride + Magnesium sulfate	
Strontium chloride + Magnesium sulfate	
Calcium chloride + Magnesium sulfate	

Equations:
$BaCl_2 + MgSO_4 \rightarrow$ _____ $+ MgCl_2$
$SrCl_2 + MgSO_4 \rightarrow$ _____ $+ MgCl_2$
$CaCl_2 + MgSO_4 \rightarrow$ _____ $+ MgCl_2$

EXPERIMENT C-6: AN INVESTIGATION OF THE PACKING OF ATOMS OR IONS IN CRYSTALS

INTRODUCTION

The solid state of matter nearly always consists of a regular arrangement of atoms, molecules, or ions. Crystal structure can be represented by a regularly repeating pattern called a space lattice. In this experiment we shall use styrofoam spheres as the building blocks and we shall study some of the ways they can be packed to form some typical metallic crystals. Three types of packing will be investigated: hexagonal packing, face-centered cubic packing; and body-centered cubic packing. We shall observe the number of nearest neighbors of the particles in each of these structures. In addition, we shall investigate some of the possibilities of packing spheres of different radii into lattices which represent ionic crystals.

MATERIALS

Styrofoam spheres (2 inches, 1 inch, and 3/4 inch diameters); short lengths of pipestem cleaners for connectors.

INSTRUCTIONS

Determine how many 2-inch spheres you can pack around a single 2-inch sphere in the same plane. Place additional spheres above and below the center sphere so that they all touch it. Connect the three groups of spheres with pipe cleaner stems so that you have the layers shown:

(1) (2) (3)

Place layer (1) on the table. Put layer (2) on top of it so that the center sphere fits closely into the depression in the center of layer (1). Then place layer (3) over the center sphere of layer (2) so that the top and bottom layers are directly over each other. Save this model of hexagonal packing for later use. If a pattern such as this were expanded into space until billions of atoms were

involved, one would have a model of a very small crystal of metallic magnesium, zinc, and many other metals. How may nearest touching neighbors does the very center atom of the middle layer have?

Construct the layers shown below just as indicated using 2-inch spheres and pipe cleaner stems as before:

(1) (2) (3)

Place layer (1) on the desk. Place layer (2) on it such that the spheres rest in the spaces between the corner spheres of the first layer. Now add layer (3) such that its spheres are directly over those in the first layer. Study this model carefully. Can you see why it is called *face-centered cubic*? This is the packing which is found in copper, silver, aluminum and many other metals.

Construct the layers shown below using 2-inch spheres. Be sure to leave space of about 1/4 inch between the spheres as shown.

(1) (2) (3)

Place the single sphere in the center of layer (1) and then place layer (3) such that its spheres are directly over the first layer. Look at the model carefully and try to see why it is called *body-centered cubic*. This type of packing is typical of alkali metals, such elements as sodium and potassium.

Ionic crystals are formed by packing positive and negative ions alternately into a lattice. We shall use 1-inch spheres for sodium ions (Na^{+}) and 2-inch spheres for chloride (Cl^{-}) in order to make a model of sodium chloride crystal. Take your model of the face-centered cubic arrangement (the second one that you constructed) and insert thirteen 1-inch spheres into the holes between the chloride ions in each layer. What type of ions surround each sodium ion? What type of ions surround each chloride ion? How many nearest neighbors of opposite charge does each sodium ion have? How many nearest neighbors of opposite charge does each chloride ion have?

The following model is the most difficult model to construct. We will use 3/4-inch spheres to represent the zinc ion (Zn^{+2}) and 2-inch spheres for the sulfide ion (S^{-2}) in order to make the crystal zinc sulfide (ZnS). Use the very *first* model you made, the *hexagonal packing model*, to represent the lattice of the larger sulfide ions. Attach one of the smaller 3/4-inch spheres, representing the

zinc ions, directly above each of the larger spheres in each of the three layers. Place the large layer on the table top with the small spheres pointed down. Place one of the small layers on the large layer in such a way that the smaller spheres fit into alternate depressions. Invert the two layers and place the other small layer, with the small spheres pointing up, above the larger layer so that each sphere on the top layer is directly above a sphere on the bottom layer. How many nearest neighbors of opposite charge do the zinc ions have?

QUESTIONS

In the ionic crystal, sodium chloride, what type of ions surround each sodium ion?

What type of ions surround each chloride ion?

How many nearest neighbors of opposite charge does each sodium ion have?

How many nearest neighbors of opposite charge does each chloride ion have?

In the zinc sulfide crystal how many nearest neighbors of opposite charge do the zinc ions have?

EXPERIMENT C-7: DETERMINING THE SIZE OF OBJECTS BY COLLISIONS

INTRODUCTION

Sometimes we wonder how it is possible to determine the size of a nucleus of an atom without actually seeing it or measuring it directly. This experiment will attempt to give you some idea of how the size of objects can be measured by indirect means. To carry out this procedure we must rely on the probability of collisions taking place. We shall determine the size of marbles in this experiment by calculating and using the probability of another marble colliding with them.

MATERIALS

Eleven marbles (10 of the same color and one of a different color); flat table; three books; meter stick.

INSTRUCTIONS

Ten marbles are placed at one end of a table in a *random* way. Some books should be placed around the edge so that the marbles being rolled will not roll off of the table. It will be helpful to place a sheet of paper under the marbles. Then mark the positions of the marbles. In that way, when the marbles have to be replaced after the collisions, their original positions can be located.

Now proceed to roll marbles, one at a time, toward the stationary marbles. Continue to roll marbles, being careful that you roll them in a *random* way, and that they cross over all points of the width labeled L in Figure C-7. Do not aim with the marbles, but merely roll them in a random manner. Each time a collision occurs replace the stationary marble. If a marble collides with more than one stationary marble, do *not* count any secondary collisions. Only record the first one. Record the number of marbles rolled, the number of collisions, and the length of the width L in the data table.

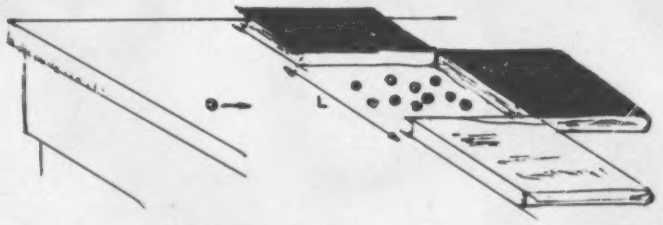


Figure C-7.

DATA

Cumulative number of marbles rolled	Collision Tallies	Cumulative Number of Collisions
10		
20		
30		
40		
50		
60		
70		
80		
90		
100		

Length of Distance L = _____ cm

Probability of a marble hitting one of the stationary marbles:

$$\text{Probability} = \frac{\text{number of collisions}}{\text{total number of marbles rolled}} = _____$$

Diameter of stationary marbles:

$$\text{Diameter} = \text{Probability} \times \frac{L}{2n} = _____$$, where n = number of stationary marbles

Actual measured diameter of marbles = _____ cm

EXPERIMENT C-8: CREATING AN ELECTRON CHARGE CLOUD ANALOGY

INTRODUCTION

If it were possible to conduct an experiment to determine the frequency of the electron's appearance at different points in the space surrounding the nucleus, these data could be plotted on a three-dimensional graph. Boundaries could be established which would outline the regions of space in which the electron could be found 95% of the time. Since it is impossible to conduct such an experiment, we will perform one by analogy. In this experiment you will determine regions of space about a bullseye in which there is a specific probability of locating a spot resulting from the impact of an object dropped from a specific distance from the target. The bullseye is analogous to the nuclear area. The object dropped is analogous to the electron.

MATERIALS

Large pieces of white paper; masking tape; ruler; dropping object (pen, pencil, or dart if available); compass; colored pencils.

INSTRUCTIONS

Make a copy of the target in Figure C-8. Tape the target to the floor so that it will not move when the object hits it. Stand on a stool or laboratory bench with the dropping object in your hand. Extend your arm so that it is about 2 meters above the target. Drop it on the target below. Repeat 99 times.

Remove the target from the floor. Locate the 95 dots which are *closest* to the bullseye and using the colored pencil, draw a smooth curve which encloses the region containing these dots. This region now represents where an electron *might* be observed 95% of the time in an experiment designed to locate it.

R_1 = 2 cm

R_2 = 4 cm

R_3 = 6 cm

R_4 = 8 cm

R_5 = 10 cm

Figure C-8.

Count the dots in each concentric circular area surrounding the bullseye. Don't forget that it must be the total cumulative number of dots which occur within that radius from the center, i.e.:

R_1 = 2 cm no. of dots = 17

R_2 = 4 cm no. of dots = 17 + 21

R_3 = 6 cm no. of dots = 38 + 20

Record the number in the data table. Staple your marked off target to the data table when you turn it in.

DATA

Number of Concentric Circles	Radii of Concentric Circles	Area of Concentric Circles	Cumulative Number of Dots in Concentric Circles
1	2 cm	12.5 cm^2	
2	4 cm	50 cm^2	
3	6 cm	113 cm^2	
4	8 cm	201 cm^2	
5	10 cm	314 cm^2	

EXPERIMENT C-9: ELECTRON PROBABILITY MAP VS. ELECTRON PATH

INTRODUCTION

Scientists are aware of all of the evidence for atoms and electrons, yet they do not claim to have proven the existence of atoms and electrons. The explanation of their attitude lies in the nature of proof. Proof requires more than consistency with all the known evidence; it requires that the proposal or explanation is and will remain the only one that can explain the facts. In this case a guarantee cannot be given. There always remains the possibility that new facts will be discovered that will change the theory; therefore scientists are unwilling to make dogmatic statements claiming *final* proof. When a scientist says he believes in atoms, he means that it is useful to believe in atoms. However, he stands ready to stop believing in atoms if new evidence were to appear that would make it no longer useful to do so. It is with this philosophy that we discuss the probability of where an electron is located in the atom.

MATERIALS

A large balloon or light weight "beach ball"; overhead projector; china marker; blindfolds; two acetate sheets.

INSTRUCTIONS

This experiment will be a group experience. A large balloon or beach ball will be tossed to you and your fellow students. The instructor will plot the "path" of the balloon as it is tossed around the room.

Then, you will be asked to blindfold each other. The balloon or ball will once again be randomly tossed around the room and the "probability map" will be plotted on the overhead projector. You will not be asked to take any data for this experiment.

EXPERIMENT C-10: A HALF LIFE MODEL OF NUCLEAR CHEMISTRY

INTRODUCTION

Physicists have developed a model of the nucleus of an atom that portrays a nucleus as a dynamic bundle of shifting energy patterns. The positively charged protons of the nucleus repel each other, and the neutrons, which are chargeless, add mass to the nucleus. The forces that bind the nucleus together are not clearly understood. The balance between the repulsion of the protons and the binding forces in the nucleus is often disturbed, resulting in the emission of a charged particle from the nucleus. This process is called radioactive decay.

Half of the atomic nuclei of a radioactive isotope in a sample, decay in a period of time known as the *half-life* of the isotope. The reason why all of the nuclei do not decay at one time is that the behavior of nuclei follows probability, as illustrated by the model of radioactive decay in this experiment. Different radioactive isotopes have different half-lives. A model for easily depicting the rate of nuclear decay is difficult to find. The use of actual radioactive samples in the determination of half-life necessitates cumbersome protective handling and sophisticated electronic instrumentation, even if these samples are readily available.

MATERIALS

Shoebox; sugar cubes; china marker; graph paper.

INSTRUCTIONS

Obtain twenty sugar cubes and an empty box with a top. Use a china marker to place a mark on *one* face of each sugar cube. Then roll the cubes into the box and put on the lid. Shake the box. After opening the box, remove the cubes that come to rest with their spotted face up. The "up" cubes represent disintegrated nuclei during the first elapsed time unit. Count and record the number of cubes left in the box. Assume that each shake represents 1000 years. Shake up the box again and remove the marked cubes that end face up. Record the data--the number of cubes remaining. Repeat this procedure fifteen times.

In order to broaden the statistical base, five trials should be conducted, each time starting with the same 20 cubes. The sum of the cubes remaining for each time interval will be recorded on the data table. Then graph the information on graph paper according to the guidelines given after the data table in Figure C-10. Obtain the half-life of this model from your graph by proceeding horizontally from the number 50 on the vertical axis until you reach the graphed line, then down to the horizontal axis to read the half-life.

DATA

Toss	Number of Cubes Remaining					Sum of Cubes Remaining
	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						

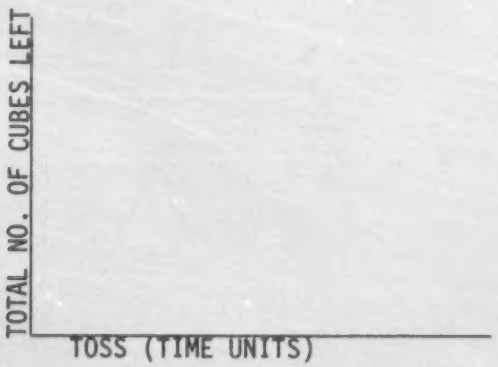


Figure C-10.

The half-life of a sugar cube in this model = _____

EXPERIMENT C-11A: COLLECTING RADIOISOTOPES FROM THE AIR

INTRODUCTION

Increasing use of radioactivity is evident everywhere. Many power plants which depend on atomic nuclear fission as a source of power are in construction or actual use. More university and industrial laboratories are making use of radioactive isotopes. Some ships use nuclear power for propulsion. Several governments are exploding nuclear fission or fusion bombs, some of which add radioactive isotopes to the air.

Some radioactivity is relatively harmless. In fact, all of use are bombarded each day with the radiation caused by cosmic rays. These rays from outer space collide with molecules in the air and break them into gragments, some of which are radioactive. With all these and other sources adding radioactive materials to the air, it is remarkable that any radioactivity-sensitive photographic paper can be manufactured, especially since eliminating radioactive contamination is difficult.

MATERIALS

A Geiger counter; a motor driven blower or vacuum pump; two felt discs; ruler; graph paper.

INSTRUCTIONS

You can collect the isotopes in the air which may be free atoms or may be absorbed on dust particles in the air by sucking air through a piece of semiporous felt. Cut a felt disc of sufficient size to cover the air intake of the blower. Cut a second felt disc the same size to serve as a control to measure background radiation. Place one felt disc over the inlet tube of the blower, place the apparatus outside. Suck air through the blower for about an hour. In the meantime place the second disc under a counting tube of a Geiger counter and obtain the counts of radioactive disintegration that occur naturally. This is called the background count. Record the data.

Now remove the disc from the pump and place it under the Geiger counter tube in exactly the same position that the first disc occupied. Obtain the counts of disintegrations over a three minute period and record the counts. Count the disintegration again over a three minute period 15 minutes later and repeat a third, fourth and fifth time at the end of 30 minutes, 45 minutes and 60 minutes respectively.

Plot your data using time on the horizontal axis and disintegration counts on the vertical axis as is indicated in Figure C-11. Don't forget to subtract the background count from each reading in order to obtain the final count. From your graph determine the half-life of the mixture of isotopes. This is the time required for the disintegrations to drop to one half the previous value.

DATA

Background Count = _____*

Trial No.	Time	Count*	Final Count* (count - background)
1	0 min.		
2	15 min.		
3	30 min.		
4	45 min.		
5	60 min.		

*All counts are to be taken for three minute intervals.

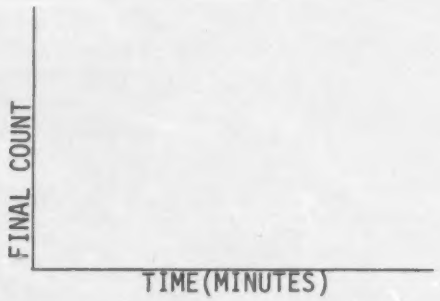


Figure C-11.

EXPERIMENT D-1: SOLIDS IN THE AIR

INTRODUCTION

The purpose of this experiment is to find the amount of dustfall in our environment. It will require thirty days to complete. Our atmosphere, even when appearing clean, is not a transparent mixture of gases. It contains varying amounts of large and small particles, some of which are dispersed from natural sources, others from human activity. The dust load in the air is not uniform from place to place or time to time.

MATERIALS

Large mouth gallon jars; tape; balance; evaporating dish; hot plate; wash bottle; stirring rod with rubber tip; insect screen; distilled water.

INSTRUCTIONS

The jar should be thoroughly cleaned and rinsed with distilled water. Pour one quart of distilled water into the jar. Place a wire screen over the top to keep insects and birds out (see Figure D-1). You should attempt to secure it firmly to the top of the jar by turning under the edges. Be sure that the screen is clean and that nothing drops into the water from the screen. Label each jar at the water level with waterproof adhesive tape and write your name, date, and purpose on the tape. Also include the name of the school and a request that the jar not be moved.

Figure D-1.

Set out the jar at the location of your choice where the measurements will be taken. Exposure should be in an open area, not under trees or eaves of a building where there is interference with the fall of material into the jar. If possible, the jar should be 5 to 6 feet above the ground to avoid contamination from coarse material blown along the ground by the wind. Freezing should be avoided. If there is a danger of freezing you may insert a clean wooden stick of finished wood long enough to reach from the bottom to the mouth of the jar. When ice forms, the presence of the stick tends to keep the jar from cracking. Soak the wood in water first for a few days to remove soluble materials.

Leave the jar exposed at the chosen location for thirty days. In a dry climate the jar should be visited every few days to insure that it does not dry out. Original water levels should be restored at these visits. In winter, use less water and check the water level in the jar after each rain. Accurate tests cannot be made if the jar runs over with water.

After thirty days, bring the jar into the laboratory and evaporate most of the water from the jar using a hot plate or water bath. Place an asbestos pad on the hot plate to protect the jar from breakage. Carefully wash down the sides and bottom of the jar with small amounts of distilled water from the wash bottle, scrubbing all surface of the inside of the jar with a stirring rod. Transfer the total sample, a little at a time, to a *weighed* evaporating dish and evaporate all the water either on a hot plate or over a water bath. Avoid overheating and splattering. When dry, cool the dish and contents, and then weigh to the nearest 0.01 gram. Subtract the weight of the empty dish to obtain the net weight of solids. During the discussion you will pool your data with the other students.

DATA

Weight of clean evaporating dish = _____ g
Weight of evaporating dish plus dried particles = _____ g
Weight of dust particles = _____ g
Observations and comments about the location where the dust particles where collected:
Class average of dust particles' weight = _____ g

EXPERIMENT D-2: THE EFFECT OF AIR POLLUTION ON VISIBILITY

INTRODUCTION

Solid and liquid particles suspended in the atmosphere absorb and and scatter light. The effect is a marked reduction in the range of visibility when the air is laden with fog, smoke, or particulate air pollutants. The extent of the scattering is influenced strongly by the number and size of particles in the air. Scattering of light is responsible for most of the decrease in the range of visibility.

MATERIALS

A map of the community; hygrometer or sling psychrometer; a good observation point; easily visible landmarks; barometer.

INSTRUCTIONS

Choose on the map a set of easily visible landmarks up to 10 miles distant. Each day note the farthest landmark which can be seen from the observation point. With each set of observations mea-

sure the relative humidity with either a hygrometer or a sling psychrometer. Discount all decreases in visibility when the relative humidity is greater than 70% as possibly influenced by fog. At the time of the visibility observations, note and record wind direction, time of day, time of year, barometric pressure, wind speed if possible, and other interesting observations, such as, types of cloud formations, etc.

DATA

Distance of farthest landmark	Rel. Hum.	Time of day	Date	Wind Dir.	Wind Speed	Baro. Press.	Other Observations

EXPERIMENT D-3: THE COMPOSITION OF AIR

INTRODUCTION

Air is a mixture of gases. We know that the separate substances composing a mixture retain their chemical properties. This provides us a means of removing individual substances in a mixture. We need only cause a reaction between the mixture and a substance that will combine with only one of the substances present in the mixture.

MATERIALS

Large beaker (600 ml); 100 ml graduated cylinder; 1-hole stopper to fit cylinder; steel wool; splints; stirring rod; tall candle; large bottle or beaker; matches; straws; watch glass; distilled water; powdered iron; ice; limewater ($Ca(OH)_2$); sodium hydroxide (NaOH) pellets; cobalt blue paper.

INSTRUCTIONS

Pour 200 ml of water into a large beaker. Secure a one-hole rubber stopper to fit a clean, 100 ml graduated cylinder. Moisten the inside of the cylinder and sprinkle the inside lightly with powdered iron. Push some loose steel wool all the way into the cylinder. Quickly insert the one-hole stopper into the cylinder and invert the cylinder in the beaker so as to trap the air in it, as shown in Figure D-3.1. Tape the apparatus so that it hangs freely.

Allow the apparatus to stand for three or four days. Note that the cylinder becomes warm to the touch. When the reaction has ceased and the cylinder has returned to room temperature, raise it if necessary, by adding water to the beaker, until the levels of the water inside and outside of the cylinder are the same. Place your finger over the hole in the rubber stopper under the water, remove the cylinder, and place it right side up on the desk, being careful to avoid losing any of the water. Thrust a glowing splint into the remaining gas in the cylinder and note the result.

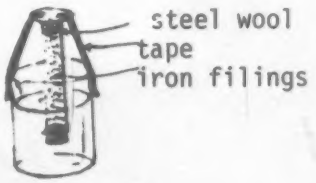


Figure D-3.1.

Remove the steel wool from the cylinder. Record on your data sheet exactly how much water is in the cylinder. Determine the volume of water that the cylinder would contain when completely filled with the stopper inserted. This is the volume of trapped air at the beginning of the experiment. Record all observations and data.

Pour 5 ml of limewater, $Ca(OH)_2$, into a small beaker and set it aside for twenty to thirty minutes. Examine at that time and note any change of appearance. Record your observations. Through a straw blow your breath into the fresh limewater for a minute or so. What gas are you exhaling? Set up a long, burning candle covered with a large bottle of air, inverted in a dish of limewater, as shown in Figure D-3.2. Make your observations and record them on the data sheet.

Stir a few pieces of ice in water contained in a beaker. Be careful not to breathe on the container or place your hands on the container. Watch the outside of the container for the collection of a liquid. Test any condensed liquid with dry blue cobalt chloride paper. What is the liquid? Place a few pellets of sodium hydroxide (NaOH) on a watch glass and leave exposed until the end of the lab period. *Do not touch the pellets with your fingers.* Observe what happens.

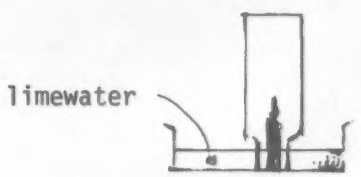


Figure D-3.2.

DATA

Percentage of oxygen in air, by volume:
1. Original volume of air (volume to the top of the cylinder) _____ ml.
2. Volume of water in cylinder after rusting _____ ml.
3. Volume of air left after rusting _____ ml.
$\%O_2 = \frac{\text{volume of water in cylinder after rusting}}{\text{total volume of cylinder}} \times 100$ $= \frac{\text{line 2}}{\text{line 1}} \times 100$ $=$ _____   %of other gases in air = $(100\% - \%O_2) =$ _____

Observations:
Limewater and air:
Limewater and exhaled breath:
Limewater and burning candle:
Conclusion:
What gas do the above limewater tests identify? _____

Observations:
Cobalt chloride test:
Sodium hydroxide and air:
Conclusion:
What gas do the above test identify? _____

Overall results:
Identified gases present in air: _____ _____ _____

EXPERIMENT D-4: SIMPLIFIED AIRBORNE SOLID SAMPLING

INTRODUCTION

There is a great deal of evidence around us today suggesting that residents who live in areas that have an air pollution problem don't give much thought to it. We inhale about 30 pounds of air each day and, with the air, varying amounts of pollutants. The particulates, finely divided solids such as smoke and dust, enter the atmosphere from the combustion of coal and fuel oil as well as incineration. The largest of these particles have a tendency to settle out of the air. When inhaled, they can hamper the performance of lung function.

MATERIALS

Cardboard pieces, hole puncher; tape; microscope; rulers; small envelopes; scissors.

INSTRUCTIONS

Cut a piece of cardboard $1\frac{1}{2}$ inches by 3 inches and fold it along the 3 inch length. Punch five holes with the puncher equally spaced. Place a long strip (five inches) of transparent tape on the table surface, gummed side up. Place the sample collector punched-hole half on the tape so that a sticky surface shows in each punched hole, as is shown in Figure D-4. Seal the top closed to prevent accidental particles from adhering to the sticky surface. Put your name on the samplers. Place the sample collectors in small envelopes which will insure that particles will be kept from the sticky surfaces until sampling is desired. Place your name on the envelope.

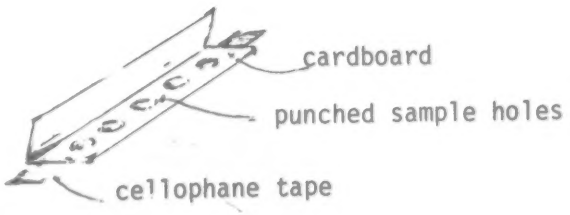


Figure D-4.

Prepare three samplers as described above. Decide where you are going to expose them and for how long. Record the locations and the time exposed on the data sheet and on the sampler itself so that no confusion will occur when examining them later. On the data sheet also note any other special conditions, i.e. wind velocity, humidity, number of hours since last rain, present weather conditions, etc. Samples can be taken in a variety of places. Use your imagination. Caution: Do not place the collector where rain will fall on it. Excessive moisture deteriorates the sampler and the gummed surface.

When the samplers are brought back to the laboratory, carefully examine the holes with a microscope under low power or with hand lens if the microscope is not available. What conclusions can you

make about the type of particles you collected and the locations from which you collected them? Record these conclusions on your data sheet.

DATA

Sample Collector No. 1	
Location:	Other Conditions:
Exposure Time Length:	
Observations made with Microscope:	
Sample Collector No. 2	
Location:	Other Conditions:
Exposure Time Length:	
Observations made with Microscope:	
Sample Collector No. 3	
Locations:	Other Conditions:
Exposure Time Length:	
Observations made with Microscope:	

Conclusions about Particles Collected
1.
2.
3.
4.
5.
6.

EXPERIMENT D-5: DESTRUCTIVE DISTILLATION OF LEAVES

INTRODUCTION

One of the seasonal tasks is the raking and collection of leaves. In the past leaves were burned at the curb. Recently, however, cities and towns have passed ordinances forbidding the burning of leaves. Ecologists encourage us to use leaves for compost piles to be used eventually as fertilizers for our gardens and lawns instead of artificial commercial fertilizers. Large cities have scheduled pickups of leaves so that homeowners will be discouraged from burning them. In some towns, tickets are issued to offenders found burning leaves. Why does it seem to be so harmful to the environment? In this experiment you will observe what happens when leaves are burned in a limited supply of air.

MATERIALS

Two clamps; ring stand; rubber stoppers (one hole and two hole); glass tubing; two large test tubes; leaves; blue litmus paper.

INSTRUCTIONS

Assemble the same apparatus that you used in Experiment B-9, *Dry Distillation of Wood.* Place the leaves in the tube on the left until it is about half filled. It might be a good idea to crush the leaves into smaller pieces. Fill the test tube on the right about one-third full of water. Make sure the short jet tube on the right has a small tip. After everything is assembled heat the test tube with the leaves gently at first. After the gas starts coming over, observe the color and compare it to the data from the sawdust distillation experiment. After the tar begins to collect under the water, try to ignite the jet tube. What color is the flame? What does the gas smell like?

After the test tube cools off, examine the remains left in the tube. Where would this residue be when burned outside? Is there a good chance of it getting into the sewer system? Examine the tar in the tube on the right. Record your observations on the data sheet. Dip a strip of blue litmus paper in the water layer of the right test tube. What happens? Describe your observations.

DATA

Observations:	
Appearance of gas	
Color of flame	
Odor	
Appearance of leave residue	
Appearance of tar	
Results of litmus test	

EXPERIMENT D-6: THE PROPERTIES OF SULFUR COMPOUNDS

INTRODUCTION

Sulfur is one of the Group VI elements on the periodic table and is therefore a member of the oxygen family of elements which consists of oxygen, sulfur, selenium, tellurium, and polonium. The properties of oxygen are sufficiently different from the rest of the elements of the family to set it apart. In fact, the last four elements of the group are sometimes referred to as the sulfur family. In this experiment various compounds of sulfur are to be studied. You have studied the element sulfur earlier in the experiment, *Allotropic Forms of Sulfur*.

MATERIALS

Roll sulfur; flowers of sulfur; copper turnings, steel wool; 6 M HCl; lead nitrate ($Pb(NO_3)_2$) paper; ammonium sulfide ($(NH_4)_2S$) solution; silver coin; concentrated sulfuric acid (H_2SO_4); hydrated copper sulfate ($CuSO_4 \cdot 6H_2O$) crystals; sucrose; distilled water; 3 M sulfuric acid; mossy zinc; copper pieces; solid sodium chloride (NaCl); potassium bromide (KBr); and potassium iodide (KI); blue litmus paper; 0.1 calcium nitrate ($Ca(NO_3)_2$); 0.1 M barium chloride ($BaCl_2$); 0.1 M strontium nitrate ($Sr(NO_3)_2$); 0.1 M lead nitrate ($Pb(NO_3)_2$); 0.5 M sodium sulfite (Na_2SO_3); dilute HCl; highly colored flower petals; apple peel; concentrated HCl; 3% hydrogen peroxide (H_2O_2); 0.1 M potassium permanganate ($KMnO_4$), solid sodium sulfite (Na_2SO_3); iron sulfide (FeS); 0.1 M cadmium nitrate ($Cd(NO_3)_2$); 0.1 M zinc nitrate ($Zn(NO_3)_2$); 0.1 M antimony trichloride ($SbCl_3$); 0.1 M cupric sulfate ($CuSO_4$); 0.1 M potassium chloride (KCl); 0.5 M ammonium sulfide ($(NH_4)_2S$); sodium hydroxide (NaOH) solution; spatula; test tubes; wood splints; small funnel tube; eyedropper; rulers; graduated cylinder.

INSTRUCTIONS

REACTIONS OF SULFUR

Place a small amount of sulfur in the bottom of each of two dry test tubes. About 2 cm above the sulfur in one tube place a loosely packed wad of copper turnings and in the other some steel wool. Heat the metal in each tube until it is red hot and then boil the sulfur. Notice any indications of a chemical reaction as the sulfur comes in contact with the hot metal. Allow the tubes to cool, remove a portion of the contents of each tube and examine. To the remainder in each test tube, add a few milliliters of dilute (6M) HCl. Test any gases evolved with moist paper that has been saturated with lead nitrate solution. Record your observations.

FORMATION OF SULFUR TARNISH

Clean a small silver coin by washing it in hot sodium hydroxide solution and then rinsing it with water. Place a few drops of ammonium sulfide, $(NH_4)_2S$, on the coin and note the formation of silver sulfide. The tarnishing of household silver is due principally to the formation of this substance from sulfur compounds in eggs, cooking gas, and perspiration. Pour 10 ml of water into a beaker and add enough sodium chloride, NaCl, to make a saturated solution. Place the tarnished coin in this solution in contact with a piece of aluminum and boil the solution. This should remove the tarnish.

REACTIONS OF SULFURIC ACID

Caution: Concentrated sulfuric acid (H_2SO_4) is extremely corrosive and should be handled carefully. Place a single small crystal of hydrated copper sulfate ($CuSO_4 \cdot 6H_2O$) into a test tube with 2 ml of concentrated sulfuric acid. Observe this crystal periodically for a half an hour after adding the acid. Record your observations.

Add about 0.5 grams of sugar (sucrose) to a test tube containing 2 ml of concentrated sulfuric acid.

Dip a wood splint into a test tube with 2 ml of concentrated sulfuric acid.

Carefully add concentrated sulfuric acid drop-wise with stirring to 25 ml of water in a small beaker. Record any noticeable heat effect. *Caution: Never pour water into acid, only acid into water.*

Pour 3-4 ml of dilute (3 M) sulfuric acid into each of two test tubes. To one test tube add a small piece of mossy zinc; to the other add a small piece of copper. Record any evidence of a reaction. *Cautiously* smell any gas evolved. Also, test the combustibility of any gas which is evolved by testing with a glowing splint. Record your observations.

Repeat the above paragraph using 3-4 ml of concentrated sulfuric acid in place of the dilute sulfuric acid. Heat *gently*.

Place 1.0 gram each of sodium chloride (NaCl), potassium bromide (KBr), and potassium iodide (KI) in separate test tubes. To each salt, *cautiously* add 1 ml of concentrated sulfuric acid. Test any gas emitted with moistened blue litmus paper and record the observations you make of each reaction.

Add 2 ml of dilute sulfuric acid to 5 ml samples of 0.1 M calcium nitrate ($Ca(NO_3)_2$), 0.1 M barium chloride ($BaCl_2$), 0.1 M strontium nitrate ($Sr(NO_3)_2$), and 0.1 M lead nitrate ($Pb(NO_3)_2$). Record any evidence of a reaction in the data table.

REACTIONS OF SULFUR DIOXIDE

Obtain two bottles of sulfur dioxide, SO_2, from a generator *under a fume hood*. Observe its color, odor, and density. (Density can be observed by trying to pour some of the gas out of the bottle.) To one bottle add about 10 ml of water and stopper. Shake. Test the liquid with litmus paper. Record your observations in the data table. Save the solution in the bottle for future use. Place a moistened brightly colored flower petal in the second bottle of SO_2. If a flower is not available, use a piece of apple peel. Notice the effect of the SO_2 on these colored pigments. Record the results. Divide the solution you were asked to save into three test tubes. To the first test tube add a few drops of concentrated hydrochloric acid (HCl), then 10 ml of 3% solution of hydrogen peroxide (H_2O_2), and finally add 5 ml of 0.1 M barium chloride solution ($BaCl_2$). Observe and record your results.

To the *second* portion of the sulfur dioxide solution, add dropwise a few ml of 0.1 M potassium permanganate ($KMnO_4$) solution. Record your observations. Test some of the resulting solution with the barium chloride solution. In preparation to using the third portion, add a piece of mossy zinc to 10 ml of dilute sulfuric acid. To this solution add dropwise the SO_2 solution from the third test tube. Note the characteristics of any solid or gas which is formed as a result. In a clean test tube add 2 grams of sodium sulfite solid to 15 ml of water in a test tube. Dissolve the solid and then add 2 ml of 0.1 M barium chloride solution. Describe the product and then add 5 ml of dilute hydrochloric acid. Record your observations.

REACTIONS OF HYDROGEN SULFIDE

Caution: Hydrogen sulfide gas is extremely toxic and should be produced only in very small quantities. To a test tube containing 2 ml of concentrated hydrochloric acid (HCl), add a single crystal of iron sulfide (FeS). Use the fume hood. Cautiously note the odor and color of the gas produced.

Place 0.5 grams of sodium sulfite (Na_2SO_3) in a test tube containing 10 ml of water. Add about 1/2 ml of dilute sulfuric acid. Take the test tube to the hydrogen sulfide generator located in the fume hood and saturate the solution in the test tube with H_2S. Describe the product which is formed.

Place 5 ml of each of the following 0.1 M solutions into separate test tubes: (a) cadmium nitrate ($Cd(NO_3)_2$), (b) lead nitrate ($Pb(NO_3)_2$), (c) zinc nitrate ($Zn(NO_3)_2$), (d) antimony trichloride ($SbCl_3$), (e) copper sulfate ($CuSO_4$), (f) potassium chloride (KCl). To each solution add a few drops of 0.5 M ammonium sulfide ($(NH_4)_2S$) solution or bubble H_2S gas through the 0.1 M solutions if a H_2S generator is available and operating. Describe each reaction completely in the data table.

DATA

Reaction of Sulfur:
With steel wool:
With copper:
Result of lead nitrate paper tests:

Observations: Sulfuric Acid Reactions	
conc. H_2SO_4 + copper sulfate	
conc. H_2SO_4 + sucrose	
conc. H_2SO_4 + wood	
conc. H_2SO_4 + water	
dilute (3 M) H_2SO_4 + zinc	
dilute (3 M) H_2SO_4 + copper	
conc. H_2SO_4 + zinc	
conc. H_2SO_4 + copper	
conc. H_2SO_4 + sodium chloride	
conc. H_2SO_4 + potassium bromide	
conc. H_2SO_4 + potassium iodide	
dilute (3 M) H_2SO_4 + calcium nitrate	
dilute (3 M) H_2SO_4 + barium chloride	
dilute (3 M) H_2SO_4 + strontium nitrate	
dilute (3 M) H_2SO_4 + lead nitrate	

Observations: Properties of SO_2 and the Sulfite Ion	
Color of SO_2	
Odor of SO_2	
Density of SO_2 relative to air	
Solubility in water	
Effect on plant pigment	
Reaction of SO_2 on plant pigment	

100

Observations: Properties of SO_2 and the Sulfite Ion (Continued)	
Acidified SO_2 solution + hydrogen peroxide + barium chloride	
Acidified SO_2 solution + potassium permanganate + barium chloride	
SO_2 solution + zinc + H_2SO_4	
Sodium sulfite + barium chloride	
Sodium sulfite + barium chloride + hydrochloric acid	

Observations: Properties of H_2S and the Sulfide Ion	
Color	
Odor	
Reaction with sodium sulfite	
Reaction of Cd^{+2} with S^{-2}	
Reaction of Sb^{+3} with S^{-2}	
Reaction of Cu^{+2} with S^{-2}	
Reaction of Pb^{+2} with S^{-2}	
Reaction of K^{+} with S^{-2}	
Reaction of Zn^{+2} with S^{-2}	

EXPERIMENT D-7: PREPARATION AND PROPERTIES OF CARBON DIOXIDE

INTRODUCTION

Carbon is the first element in the fourth group of the periodic table. Carbon dioxide is an essential compound in nature since it is necessary for photosynthesis. It is also a product of all combustion processes. In addition, carbon dioxide is an important part of many industrial processes. It also has many practical everyday uses, as in refrigeration, beverages, fire extinguishers, etc. Recently the amount of carbon dioxide present in the air has become a point of heated debate for ecologists.

MATERIALS

Wide mouth bottles; pneumatic trough; wood splints; candle; matches; graduated cylinder; chalk; watch glass; glass tubing; rubber tubing; funnel tube; glass squares; concentrated hydrochloric acid (HCl); marble chips ($CaCO_3$); distilled water; limewater ($Ca(OH)_2$); limestone pieces.

INSTRUCTIONS

Set up the apparatus as shown in Figure D-7 for the preparation of carbon dioxide. Fill up three collection bottles with water and invert them in the trough. Cover the bottom of the generator bottle with marble chips ($CaCO_3$) and add enough water through the funnel tube to cover the marble chips and the end of the funnel tube. Then add enough concentrated hydrochloric acid (HCl) to produce a steady evolution of gas; add acid from time to time to maintain a steady flow of gas. Discard the first bottle of gas collected as impure, and collect three bottles of the carbon dioxide being generated. Cover the bottles with glass squares and set them upright on the table. Do not dismantle the apparatus until the end of the experiment.

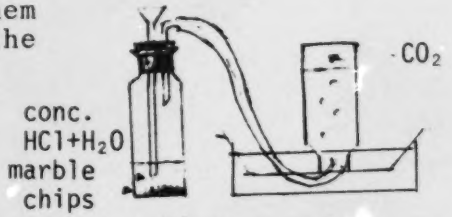


Figure D-7.

What is the color and odor of carbon dioxide? Pour about 10 ml of limewater ($Ca(OH)_2$) into a test tube. Let the carbon dioxide (CO_2) from the generator bubble through the limewater. This is a test for the presence of carbon dioxide. Describe any change observed. Next, add about 20 ml of water to a bottle of carbon dioxide, cover the bottle quickly with the palm of the hand and shake vigorously. Describe the solubility of carbon dioxide in water. To test for the presence of carbon dioxide in the air, pour a few ml of limewater in a watch glass and set it aside until the end of the period. Lower a short lighted candle in a small beaker. Into this beaker pour (as you would pour water) one of the bottles of carbon dioxide. What is the result? What can you say about the density of carbon dioxide? Does it support combustion?

Burn a wood splint, paper, candle or other organic substance in a bottle of air. Add 10 ml of limewater, cover the bottle, and shake. Record your observations and conclusions on the data table. Exhale through a straw into 5 ml of limewater in a test tube. Record your observations. Finally, examine the watch glass with limewater that was set aside earlier in this experiment.

Rinse out the generator and place in it some chalk or a piece of limestone. Fit the stopper and add a little water; follow with a little concentrated hydrochloric acid through the funnel tube. Pass the gas generated into 5 ml of limewater in a test tube. What results do you observe?

Dismantle the apparatus only so far as to wash out the bottle and tubes. Do not dismantle the tubes, rubber hosing, and rubber stopper as they will be used in Experiment D-8, *Three Oxides of Nitrogen.*

DATA

Observations: Carbon Dioxide
Color
Odor
Limewater test
Solubility in water
Density
Combustion (state your evidence)
What happened when you tested the results of burning an organic substance?
What conclusion does this bring you to?
Do you exhale CO_2? How do you know?
What happened to the limewater which stood exposed to the air in the lab?
Do chalk or limestone liberate CO_2 just like marble chips?

Reaction Equation:						
Calcium Carbonate	+ Hydrochloric Acid →	Carbon Dioxide↑	+ Water +	Calcium Chloride		
$CaCO_3$	+ _____ →	_____↑	+ ____ +	_____		
(don't forget to balance the equation!)						

EXPERIMENT D-8: THREE OXIDES OF NITROGEN

INTRODUCTION

Nitrogen from Group V of the Periodic Table forms five different oxides. Two are unstable. In this experiment we will study three stable oxides: nitric oxide (NO), nitrogen dioxide (NO_2), and nitrous oxide (N_2O).

MATERIALS

Florence flask; funnel tube; glass tubing; rubber tubing; wide mouth bottles; burner; one-hole stopper; matches; wood splints; balance; graduated cylinder; pneumatic trough; evaporating dish; concentrated nitric acid (HNO_3); distilled water; copper pellets or turnings; ammonium nitrate (NH_4NO_3).

INSTRUCTIONS

Set up a flask generator as is shown in Figure D-8 upon a wire gauze on a ring and ring stand. Place 5 grams of copper in the flask. Dilute 20 ml of concentrated nitric acid by pouring it into 20 ml of distilled water and then pour the diluted acid down the funnel tube. Add enough of it to cover the lower end of the funnel tube. Warm *gently, only if necessary*. Collect three bottles of the nitric oxide over water. Be careful to keep air out of the bottles by making sure that the wide mouth bottles are full to the top of water before inverting in the trough. Note the color of the nitric oxide and, from the way you have collected it, consider its solubility in water. When you have finished collecting the gas, add water through the funnel tube.

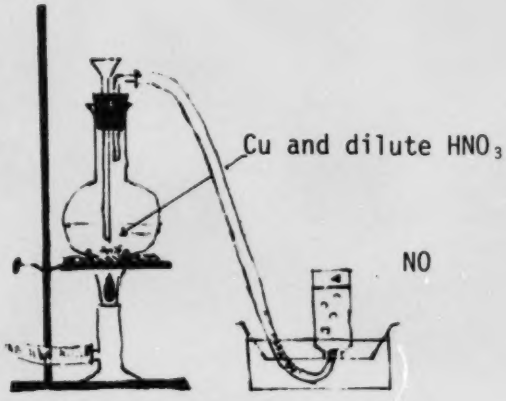


Figure D-8.

Lift one of the bottles of nitric oxide (NO) that you just prepared and collected out of the water so that the gas comes in contact with the air. Observe any changes. Nitrogen dioxide (NO_2) is being formed. It is a spontaneous reaction. Cautiously observe its odor. Immediately invert the bottle of NO_2 in a beaker of water. Observe and consider its solubility. Test another bottle of nitric oxide with a blazing splint.

Set up a glass Pyrex test tube generator fitted with a one-hole stopper and a delivery tube. Place 10 grams of ammonium nitrate (NH_4NO_3) in the tube. *Caution: Ammonium nitrate may become explosive when heated too strongly. Heat it gently and do not decompose more than about half of the ammonium nitrate.* Do not let the melted ammonium nitrate foam too much. Collect two test tubes of the gas by water displacement. Observe the color of nitrous oxide (N_2O). Also note its color. Test it with a glowing splint.

DATA

Name of Oxide	Formula	Solubility in Water	Odor	Color	Combustibility
Nitric Oxide					
Nitrogen Dioxide					
Nitrous Oxide					

EXPERIMENT D-9: LEAD AND ITS COMPOUNDS

INTRODUCTION

Lead is an example of a heavy metal. It has a wide variety of uses: paints, gasolines, storage batteries, and pipes. The aim of this experiment is to observe some of its compounds.

MATERIALS

Lead pieces; zinc strips; dilute nitric acid (HNO_3); dilute hydrochloric acid (HCl); dilue sulfuric acid (H_2SO_4); 0.2 M potassium chromate (K_2CrO_4) solution; saturated hydrogen sulfide (H_2S) solution; 0.2 M sodium sulfate (Na_2SO_4) solution; concentrated hydrochloric acid (HCl); glycerin; lead dioxide (PbO_2); lead monoxide (PbO); sandpaper; balance; graduated cylinder.

INSTRUCTIONS

Obtain a piece of lead and sandpaper it. Note as many physical properties as possible.

Place 1 gram of lead in a beaker and add 20 ml of dilute nitric acid (HNO_3). Boil for 15 minutes and add 40 ml of water. Divide into six portions. If the mixture boils almost to dryness before 15 minutes are up, add a little more of the dilute nitric acid. Add to each of the six portions in order the specified amount of the following reagents (for example to the first portion add the HCl). First portion: 5 ml dilute HCl, second portion: 2 ml di-

lute sulfuric acid (H_2SO_4), third portion: 5 ml potassium chromate (K_2CrO_4), fourth portion: saturated H_2S solution until a solid forms, fifth portion: solution of sodium sulfate (Na_2SO_4) until solid forms, sixth portion: piece of zinc solid for 5 minutes. Record all observations in the data table. Then put each of the portions first in a cold water bath and then heat them to boiling in order to determine thc differences in their solubility in cold and hot water. Record any observations. *Caution: All soluble lead compounds are poisonous. Use care when disposing of them into a chemical crock. Do not pour them down the drain.*

Obtain 0.5 gram samples of lead monoxide and lead dioxide. Write the formulas and a brief observation for these compounds. Test their solubility in dilute nitric acid (HNO_3), and warm if necessary. Record your observations. To another sample of each oxide add concentrated hydrochloric acid (HCl). Record what happens to each oxide.

Optional : Mix PbO (litharge) with glycerin until a stiff paste is formed. Mold it into any shape you wish to cast and allow it to stand until it sets. It should form a very hard waterproof mass. You can use it to cement two objects together. It is commonly called "Plumber's Cement."

DATA

Physical Properties of Lead
Color
Hardness
Density

Reactants	Observations	Cold Water Bath	Hot Water Bath
Lead Solution and 5 ml HCl			
Lead Solution and H_2SO_4			
Lead Solution and K_2CrO_4			
Lead Solution and H_2S			
Lead Solution and Na_2SO_4			
Lead Solution and Zn			

EXPERIMENT D-10: SOME PROBLEMS OF INCINERATION

INTRODUCTION

In order to dispose of garbage and of flammable rubbish, an immediate and apparently simple solution to the problem is, "Burn it." In actual practice, however, burning is not the complete answer, especially if air pollution is to be avoided. For many types of materials, burning does not proceed as simply as does the burning of a stick of wood. Some materials melt before they burn and form a puddle in the incinerator. Old rubber tire and rubberlike materials may burn incompletely, causing huge volumes of smoky soot.

MATERIALS

China marker; timer; balance; matches; burner; ring and ringstand or tripod; wire screen; samples of: paper, plastic, food wrappings, fabrics, cardboard, foil, paper wrappings, tin cans with labels attached, and newspapers.

INSTRUCTIONS

Take 2-inch square samples, one at a time, of the items listed above. Weigh each one. Write in the data table a brief description with respect to color, moisture content, thickness, and other identifying characteristics.

Center the sample on the wire screen which is in turn centered on the ringstand. The position of the stand is marked on the table top and the lighted burner is placed so that it is centered beneath the sample. The burner is lighted and turned on to the same position of the gas supply throughout the experiment. In performing the experiment the burner is moved into place and the timer started. All changes are to be observed and recorded. The time is noted and recorded when no more changes take place in the material being incinerated. If smoke is emitted from the sample, determine whether or not a second burner will burn it.

DATA

Description of Sample	Weight	Condition	Time Heating Started	Time Heating Ended	Obser. During Heating	Cond. of Res after Heat.

EXPERIMENT D-10A: TOTAL ACIDS IN AIR

INTRODUCTION

The main idea of this experiment is to determine the acid content of air, using a paper probe on the end of a glass tube. The paper is soaked in an indicator solution. When air that contains acids is drawn through the probe, the indicator changes color. The time required for this event to occur after the air flow is started is a measure of the acid concentration in the air sample. In addition to SO_2, contaminated air may contain sulfuric acid as a mist, oxides of nitrogen which form acids when they combine chemically with water, and some solids in dust which act as acids when they dissolve in water. The problem is to determine the extent of such contamination.

MATERIALS

Funnel tube; aspirator or vacuum pump; rubber tubing; paper toweling; timer; stirring rod; wire screen if needed; various household sprays; sodium hydrogen carbonate ($NaHCO_3$) solution; methyl orange indicator solution; dilute hydrochloric acid (HCl); glycerin.

INSTRUCTIONS

The first trial is a test for practice and to see that everything is working properly. The probe is prepared by placing wet-strength white paper toweling cut to fit on the end of a thistle or funnel tube. It can be held down with a rubber band (see Figure D-10A). If it is found that the paper when moist does not withstand the flow of air through it without rupturing, support it by a perforated porcelain disc or by a wire screen cut to fit over the opening. A drop of glycerin is added to the paper to avoid its drying. One drop of the indicator (use a stirring rod to place the drops) and one drop of the sodium bicarbonate buffer are also placed on the paper. More drops may be needed if the color is not apparent. If the paper on the probe will not absorb this quantity of liquid without drooping, start again but use two or more thicknesses of paper on the probe. Start the air pump and the timer. The end point is reached when a red color appears. With the buffer present, carbon dioxide has little effect on the indicator but sulfur diox-

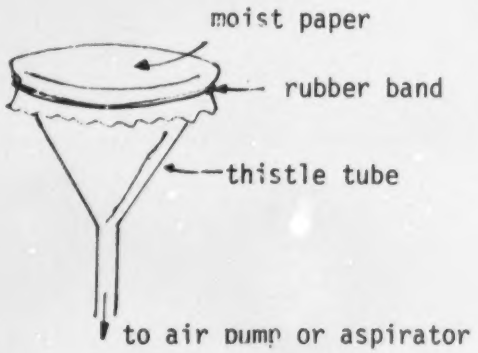


Figure D-10A.

ide and other stronger acids in water do make a change. In this preliminary trial test, however, draw air from over the top of an open bottle of dilute hydrochloric acid. The change in color should occur quite soon. When the change occurs, stop the timer and calculate the elapsed time.

Now make a trial of air of the chemical laboratory. If a movable air pump is available, test other locations, i.e. outside, the hallway, near the incinerator, the air intake duct, exhaled breath, and common household sprays. However, it is important that the same procedure, once established, be followed each time. Then the length of time needed for the change is a measure of the relative acidity of the air sampled.

DATA

Air Sample Location	Time Start	Time Stop	Time Elapsed	Color of Probe
HCl				
Air in Lab				
Exhaled Breath				
Outside Air				
Forced Air Input Into Room				
Weed Killer				
Aerosol Hair Spray				
Aerosol Air Freshener				
Spray Deodorant				
Spray Bomb Insecticide				

EXPERIMENT D-11: ANALYSIS OF SOIL FOR SOME COMMON CONSTITUENTS

INTRODUCTION

Few valuable plants can thrive in a soil which is strongly acid. Unfortunately, because of weathering of the soil, or removal of soil constituents by crops, the basic constituents are removed more quickly than the acid constituents. The important metallic or basic constituents are compounds of Na, K, N, Mg, Ca, Fe, Al, and NH_4^+. The important nonmetals which form acid radical or acid constituents are C, Cl, S, Si and P.

MATERIALS

Soil; distilled water; red and blue litmus paper; platinum or nichrome wire for flame test; saturated ammonium oxalate ($(NH_4)_2C_2O_4$) solutions; Nessler's solution; 10% potassium thiocyanate (KSCN) solution; soda lime; Titan-yellow solution (0.15%) solid calcium hydroxide ($Ca(OH)_2$); 0.08 M sodium cobaltinitrite solution ($Na_2Co(NO_2)_6$); 6 M ammonium hydroxide; 12 M hydrochloric acid (HCl); 12 M nitric acid (HNO_3); 0.5 M silver nitrate ($AgNO_3$); 0.25 M barium chloride ($BaCl_2$); 0.5 M ammonium molybdate ($(NH_4)_2MoO_4$); 5% diphenylamine solution; 3 M sodium hydroxide (NaOH); balance; filter paper; evaporating dish; thermometer.

INSTRUCTIONS

Analysis for Moisture: Weigh an evaporating dish. Place 50 grams of the pulverized soil sample in the evaporating dish and weigh accurately. Determine the weight of the sample by subtracting the weight of the evaporating dish from the total weight of the dish and the sample. The dish and contents should be heated at about 105°C for a total of about 90 minutes. Let the dish cool and then weigh again. The loss in weight is the moisture that was in the sample. Save this sample.

Analysis for Humus: Heat the sample from the *Analysis for Moisture* over the blue Bunsen burner flame until all of the dark color disappears. Cool and weigh again. The loss in weight is due to the removal of the organic matter, or humus, in the sample.

Prepare a new sample of soil for testing for nonmetallic minerals in the following way: To a 50 gram sample of the soil, add 100 ml of distilled water and 1 gram of calcium hydroxide ($Ca(OH)_2$). Mix thoroughly by shaking. Filter and save the filtrate. Perform the following tests and record your observations:

Test for Nitrates: Add 15 to 20 drops of the filtrate just prepared to about 5 ml of diphenylamine solution. A dark blue color indicates the presence of a nitrate.

Test for Ammonia and for Organic Nitrogen Compounds: Add 1 ml of Nessler's solution to about 5 ml of the prepared filtrate and warm gently. The presence of ammonia or nitrogen compounds is indicated by a yellow color. Another confirmatory test for the ammonia is to mix a sample of soil with soda-lime in a test tube. Heat gently and test the fumes with moist red litmus paper. A blue color of the litmus indicates the presence of ammonia.

Test for Sulfates: To 5 ml of the prepared filtrate add 1 ml of 0.25 M barium chloride ($BaCl_2$) solution. If a white precipitate of $BaSO_4$ forms which is insoluble in 6 M hydrochloric acid (HCl), the sulfate ion may be present.

Test for Chlorides: To 5 ml of the prepared filtrate which has been solidified by adding 2 drops of 6 M nitric acid (HNO_3), add 1 ml of 0.5 M silver nitrate ($AgNO_3$) solution. If a white precipitate (AgCl) forms, which is soluble in ammonium hydroxide (NH_4OH) but insoluble in nitric acid, the chloride ion is present.

Test for Phosphates: Add concentrated nitric acid (HNO_3) to 10 ml of the prepared filtrate until the filtrate reacts acidic to litmus. Dilute with distilled water and add 5 drops of 0.5 M ammonium molybdate ($(NH_4)_2MoO_4$) solution. Warm and let stand. A yellow precipitate indicates the presence of phosphate ions.

Test for Carbonates: To 1/4 teaspoonful of soil add 10 drops of dilute (1:3) hydrochloric acid (HCl). An evolution of a gas indicates the presence of carbonates. The gas would be carbon dioxide.

To prepare a second batch of soil, add 50 ml of distilled water to 25 grams of the soil sample, shake the misture thoroughly and filter.

Test for Calcium Ions: Heat about 5 ml of the filtrate to boiling. Add 2 ml of concentrated ammonium oxalate ($(NH_4)_2C_2O_4$) solution. Calcium ions in soluble compounds are indicated by a white precipitate of calcium oxalate, CaC_2O_4.

Tests for Potassium and Sodium Ions: Add about 3 grams of soil to 15 ml of distilled water. To this add 5 ml of concentrated hydrochloric acid (HCl). Boil and filter. Now add a few drops of 0.08 M sodium cobaltinitrite ($Na_3Co(NO_2)_6$) solution to one half of the filtrate. A light yellow precipitate indicates the presence of potassium. Evaporate a portion of the filtrate to

dryness, and apply a flame test to the residue. This is done by dipping a clean piece of platinum or nichrome wire in the dry filtrate and placing it in a flame. The sodium ion is indicated by a yellow tinge to the flame.

Test for Magnesium Ions: Add a few drops of a 10% solution of sodium hydroxide (NaOH) to 5 ml of the filtrate prepared above in the test for calcium ions. A peach-pink solution with a fine pink precipitate indicates magnesium when a few drops of Titan-yellow solution are added with stirring.

Test for Iron Ions: Add ammonium hydroxide (NH_4OH) to 10 ml of the prepared soil filtrate until it shows basic action at litmus. Heat to boiling and filter. Save the residue in the filter paper. Add 2 or 3 ml of dilute hydrochloric acid (HCl) to the residue in the filter paper and test the filtrate with a few drops of 10% potassium thiocyanate (KSCN) solution. The presence of iron ions is indicated by a red color.

DATA

Analysis of Moisture and Humus
Moisture
Weight of dish = _____ grams
Weight of dish and soil before drying = _____ grams
Weight of dish and soil after drying = _____ grams
Loss in weight of the sample = _____ grams
Percent of moisture = _____ %
Humus
Weight of dish and soil before heating = _____ grams
Weight of dish and soil after heating = _____ grams
Weight of humus = _____ grams
Percent of humus = _____ grams

Analysis for Non-Metallic Ions:			
	Present	Not Present	Observations
Nitrogen			
Ammonia-Nitrogen			
Chloride			
Phosphate			
Carbonate			
Sulfate			

Analysis for Metallic Ions:			
	Present	Not Present	Observations
Calcium			
Potassium			
Sodium			
Magnesium			
Lead			

EXPERIMENT D-12: MINERAL CONTENT OF A FERTILIZER

INTRODUCTION

The correction of soil deficiencies is usually made by adding the necessary substances to the soil. This is commonly done by adding a fertilizer to the soil in order to enrich its mineral content.

MATERIALS

Commercial fertilizer; glacial acetic ($HC_2H_3O_2$) acid (1:3); funnel; filter paper; graduated cylinder; balance; all of the solids and solutions listed with Experiment D-11, *Analysis of Soil for Some Common Constituents.*

INSTRUCTIONS

Thoroughly shake 50 grams of fertilizer with 100 ml of the glacial acetic ($HC_2H_3O_2$) acid (1:3) and filter. Test the filtrate obtained in the following manner and record your results on the data sheet:

Test for Nitrates: Add 15 to 20 drops of the filtrate just prepared to about 5 ml of diphenylamine solution. A dark blue color indicates the presence of nitrate ions.

Test for Ammonia and for Organic Nitrogen Compounds: Add 1 ml of Nessler's solution to about 5 ml of the prepared filtrate and warm gently. The presence of ammonia or nitrogen compounds is indicated by a yellow color. Another test for the ammonia is to mix a sample of fertilizer with soda-lime in a test tube. Heat gently and test the fumes with moist red litmus paper. A blue color of the litmus indicates the presence of ammonia.

Test for Chlorides: To 5 ml of the prepared filtrate which has been acidified by adding 2 drops of 6 M nitric acid (HNO_3), add 1 ml of 0.5 M silver nitrate ($AgNO_3$) solution. If a white precipitate (AgCl) forms, which is soluble in ammonium hydroxide (NH_4OH) but insoluble in nitric acid (HNO_3), the chloride ion is present.

Test for Sulfates: To 5 ml of the prepared filtrate add 1 ml of 0.25 M barium chloride ($BaCl_2$) solution. If a white precipitate of $BaSO_4$ forms, which is insoluble in 6 M hydrochloric acid (HCl), the sulfate ion may be present.

Test for Phosphates: Add concentrated nitric acid (HNO_3) to 10 ml of the prepared filtrate until the filtrate reacts acidic to litmus. Dilute with distilled water and add 5 drops of 0.5 M ammonium molybdate ($(NH_4)_2MoO_4$) solution. Warm and let stand. A yellow precipitate indicates the presence of phosphate ions.

Test for Carbonates: To 1/4 teaspoonful of fertilizer add 10 drops of dilute hydrochloric acid (1:3). An evolution of a gas indicates the presence of carbonates. The gas would be carbon dioxide.

Prepare a second batch of fertilizer by adding 50 ml of distilled water to 25 grams of the fertilizer sample. Shake the mixture thoroughly and filter.

Test for Calcium Ions: Heat about 5 ml of the filtrate to boiling. Add 2 ml of concentrated ammonium oxalate ($(NH_4)_2C_2O_4$) solution. Calcium ions in soluble compounds are indicated by a white precipitate of calcium oxalate, CaC_2O_4.

Tests for Potassium and Sodium Ions: Add about 3 grams of fertilizer to 15 ml of distilled water. To this add 5 ml of concentrated hydrochloric acid. Boil and filter. Now add a few drops of 0.08 M sodium cobaltinitrite ($Na_3Co(NO_2)_6$) solution to one half of the filtrate. A light yellow precipitate

indicates the presence of potassium. Evaporate a portion of the filtrate to dryness, and apply the flame test to the residue. The sodium ion is indicated by a yellow flame.

Test for Magnesium Ions: Add a few drops of a 10% solution of sodium hydroxide (NaOH) to 5 ml of the filtrate prepared above in the test for calcium ions. A peach-pink solution with a fine pink precipitate indicates magnesium when a few drops of Titan-yellow solution are added with stirring.

Test for Iron Ions: Add ammonium hydroxide (NH_4OH) to 10 ml of the prepared fertilizer filtrate until it shows basic action to litmus. Heat to boiling and filter. Save the residue in the filter paper. Add 2 or 3 ml of dilute hydrochloric acid (HCl) to the residue in the filter paper and test the filtrate with a few drops of 10% potassium thiocyanate (KSCN) solution. The presence of iron ions is indicated by red color.

DATA

Analysis for Non-Metallic Ions:			
	Present	Not Present	Observations
Nitrates			
Ammonia-Nitrogen			
Chlorides			
Sulfates			
Phosphates			
Carbonates			

Analysis for Metallic Ions:			
	Present	Not Present	Observations
Calcium			
Potassium			
Sodium			
Magnesium			
Iron			

EXPERIMENT D-13: THE ANALYSIS OF SUBSTANCES CONTAINED IN PLANT TISSUE

INTRODUCTION

Ten elements have been found to be essential for the growth of plants. These elements are found in various chemically combined forms (compounds) in plants and in soil. The average plant obtains about 9.5% of its weight from the air, 89% from water, and 1.5% from minerals in the soil.

MATERIALS

Various beakers; oven; test tubes; crucible; burner; red litmus paper; cobalt blue glass; nichrome or platinum wires; balances; graduated cylinder; matches; centigrade thermometer; finely divided leaves and plant fragments; concentrated nitric acid; concentrated ammonium hydroxide; concentrated hydrochloric acid; soda lime; dilute hydrochloric acid (1:3); 0.2 M silver nitrate solution; 0.25 M barium chloride; 0.5 M ammonium molybdate; 10% potassium thiocyanate; concentrated ammonium oxalate; 1 M sodium orthophosphate.

INSTRUCTIONS

Analysis for Water: Carefully dry a 250 ml beaker and label it with your name. Fill the beaker with finely divided leaves and plant fragments. Weigh the filled beaker accurately. Place the beaker and plant material in an oven where the temperature can be kept slightly above 100°C for 5 to 6 hours. When cool, weigh again, and record the weight in your data table. Repeat the heating for several hours and weigh again after cooling. When a fairly constant weight is obtained, calculate the percent of water in the plant tissue.

Analysis for Carbon, Hydrogen and Oxygen: Place a small portion of the dried plant material in a test tube and heat strongly in the flame of your burner. Make observations and observe if any liquid forms on the sides of the tube. Also examine the color of the residue in the tube. Record you conclusions on the data table.

Fill a large crucible full of the *dried* plant material and heat strongly over your burner until the remains are completely burned to a grayish ash. Divide the ash into 0.5 gram portions.

To a fresh portion of plant ash (0.5 gram) add 5 ml of distilled water and 5 ml of concentrated nitric acid (HNO_3). Heat to boiling, add 10 ml of distilled water and filter. Divide the filtrate into three parts.

Analysis for Chlorine: To one portion of the filtrate add 2 ml of 0.2 M silver nitrate ($AgNO_3$) solution. A white precipitate of silver chloride (AgCl) proves the presence of a chlorine compound. This precipitate is insoluble in nitric acid and soluble in ammonium hydroxide.

Analysis for Sulfate: To the second portion of the filtrate add 2 ml of 0.25 M barium chloride ($BaCl_2$) solution. A white precipitate shows the presence of a sulfate and therefore an original sulfur compound.

Analysis for Phosphate: To the third portion of the filtrate add 5 ml of 0.5 M ammonium molybdate ($(NH_4)_2MoO_4$) solution and heat to about 40°C. A yellow precipitate, on standing, shows the presence of a phosphate and therefore an original phosphorus compound in the plant.

To a second portion (0.5 gram) of the plant ash add a little distilled water, about 5 ml of concentrated hydrochloric acid (HCl), and a little concentrated nitric acid (HNO_3) (2 ml). Boil this mixture until evaporated nearly to dryness. Dilute the remaining solution with 15 ml of distilled water. Filter, save the filtrate, and note any insoluble residue which indicates silicon compounds.

Analysis for Iron: Make the filtrate from above basic to litmus by adding an excess of ammonium hydroxide. Heat to boiling and filter to remove the precipitate formed. Save the filtrate. Add 3 ml of dilute (1:3) hydrochloric acid (HCl) to the residue on the filter paper and save this filtrate. To this second filtrate, add a few drops of 10% potassium thiocyanate solution (KSCN). A red color indicates the presence of iron compounds in the plant. Record all of your observations on your data sheet.

Analysis for Calcium: To the first filtrate from the test for iron above add heat until it boils. Then add 2 ml of ammonium oxalate ($(NH_4)_2C_2O_4$) solution. A white precipitate shows the presence of calcium compounds in the plant. Filter the solution and save the filtrate for the magnesium test below.

Analysis for Magnesium: Add 5 ml of distilled water to the filtrate saved from the calcium test above. Divide the solution into two parts. Save one part for the sodium and potassium tests below. To the other portion add 5 ml of 1 M sodium phosphate (Na_3PO_4) solution slowly drop by drop and add 5 ml of concentrated ammonium hydroxide (NH_4OH). If no change is evident, set aside for a time. A white precipitate indicates the presence of magnesium compounds.

Analysis for Sodium and Potassium: Evaporate the portion of filtrate saved from the above test for magnesium to dryness. Then heat strongly until fuming ceases. Allow to cool. To the residue add a few drops of concentrated hydrochloric acid (HCl). Apply the flame test for sodium and potassium. Remember to use a piece of cobalt blue glass for sodium and potassium. Positive reactions in the flame (yellow for sodium and purple for potassium) indicate the presence of sodium and potassium compounds in the plant. Record your results on the data sheet.

Analysis for Nitrogen Compounds: Mix about 1 gram of the dried plant materials with 10 grams of soda lime, which is a mixture of sodium hydroxide and calcium hydroxide. Place this mixture in a Pyrex test tube and heat strongly. Test the evolved gases with moist red litmus paper and note the odor of the fumes. Record all observations on your data sheet. The odor may indicate ammonia, and the litmus paper test may prove its presence. The formation of ammonia is a result of the decomposition of more complex nitrogen compounds in the tissues of the plant.

DATA

Analysis for Water:	
Weight of beaker and plant material before heating	g
Weight of empty beaker	g
Weight of plant material before heating	g
Weight of beaker and dried plant material	g
Weight of dried plant material	g
Weight of water in plant material [wt. of plant material before heating]-[wt. of dried plant material]	g
Percent of water in plant material*	%

$$* \frac{\text{wt. of water in plant material}}{\text{wt. of plant material before heating}} \times 100$$

Analysis for Various Elements:			
	Present	Not Present	Observations
Carbon			
Hydrogen			
Oxygen			
Chlorine			
Sulfur			
Phosphorus			
Iron			
Calcium			
Magnesium			
Sodium			
Potassium			
Nitrogen			

EXPERIMENT D-14: CHEMICAL PROPERTIES OF WATER

INTRODUCTION

Water is a very stable substance that does not decompose easily. It does react, however, with a great many substances. Water is necessary for the formation of some crystals and is present in such crystals as water of hydration. Water promotes many chemical changes.

MATERIALS

Balance; beakers; wide mouth bottle; burner; test tube holder; evaporating dish; Erlenmeyer flask (any size); glass squares; graduated cylinder; deflagrating spoon; test tubes (at least one Pyrex); watch glasses; splints; stirring rod; spatula; calcium metal; calcium oxide; copper (II) sulfate ($CuSO_4$) crystals; red and blue litmus paper; red phosphorus; potassium metal; sodium aluminum sulfate ($NaAl(SO_4)_2$) powder; sodium hydrogen carbonate ($NaHCO_3$); sodium metal; crystals of magnesium chloride, potassium nitrate, sodium carbonate, sodium sulfate, barium chloride, cobalt (II) chloride, iron (II) sulfate, nickel (II) sulfate, potassium chlorate, potassium chloride, potassium sulfate, sodium chloride, and anhydrous calcium chloride; electrolysis apparatus; power supply; distilled water; concentrated sulfuric acid (H_2SO_4); phenolphthalein solution.

INSTRUCTIONS

Drop a small piece of calcium metal into a test tube which is one quarter full of water. Observe the results. Perform the flaming splint test for hydrogen. Dip a stirring rod into the liquid in the test tube and touch it to a piece of red litmus paper. Record all observations.

Place a lump of calcium oxide (CaO) on a glass plate. Pour about 1 ml of water on the calcium oxide, until it becomes quite wet. It may take a minute or two for the reaction to occur. After the reaction is over, transfer some of the residue on a stirring rod or spatula to a piece of moist red litmus paper which is on a clean glass plate. Record all your observations on the data sheet.

Mix 1.0 gram of powdered sodium aluminum sulfate ($NaAl(SO_4)_2$) and 2.0 grams of sodium hydrogen carbonate ($NaHCO_3$) on a piece of paper, and then add the mixture to a dry Erlenmeyer flask. Is there any evidence of chemical action? Pour 30 ml of water into the flask. What do you observe? These substances are used in some types of baking powders. Describe what you observe.

Place a crystal of copper (II) sulfate in a dry Pyrex test tube, and heat the test tube gently with a low burner flame. Hold the test tube with the open end inclined slightly downward. As the reaction proceeds you should be able to tell why this is necessary. Observe the substance which appears near the open end of the test tube. After the test tube cools, add a few drops of water to the residue in the test tube. What results from this action? Describe everything on the data sheet.

In *dry* test tubes, heat successively a few small crystals of the salts listed in the data table. Tabulate the results. In the middle column indicate whether or not water of hydration was present. Use cobalt chloride paper to test for the presence of water. In the column at the right of the data table, describe the residue, using appropriate terms like: melts, unchanged, forms powder, etc.

Place a spatula of each of the following salts on separate watch glasses: sodium carbonate, sodium sulfate, magnesium chloride, calcium chloride, and potassium nitrate. As quickly as possible, determine the accurate mass of each watch glass and contents by weighing them on a balance. Record this data. Set the watch glasses aside to remain undisturbed until the next laboratory period. Inspect them from time to time during this laboratory period to note changes in appearance. At the beginning of the next laboratory period, weigh each watch glass and contents again and record these new weights. Determine whether there has been a gain or loss of weight.

TEACHER DEMONSTRATION

Cut a piece of sodium the size of a grain of rice from a larger chunk of the element. Examine the freshly cut surfaces. Drop the small piece into a large beaker which is half-filled with water containing several drops of phenolphthalein. Repeat the procedure using a small piece of potassium. Note the relative reactivity of sodium and potassium. The gas evolved in both reactions is hydrogen.
Caution: Sometimes a slight explosion scatters particles of sodium and potassium so be certain to stand aside during the reactions.

Place a very small amount of red phosphorus in a clean combustion or deflagrating spoon and ignite in a Bunsen burner flame. Lower the spoon into a wide-mouth bottle containing 5-10 ml of distilled water. Cover as completely as possible with a glass square. After burning has ceased, remove the spoon and vigorously shake the contents of the bottle. The white smoke is an oxide of phosphorus. Test the solution with litmus and record your observations.

Set up the electrolytic apparatus as shown in Figure D-14. To about 50 ml of distilled water, add *slowly* 3-4 ml of concentrated sulfuric acid. Pour this mixture into the apparatus. Use a *direct current* of about 5-10 volts so that bubbles of gas evolve rapidly. Allow the reaction to continue until there are at least 20 ml of gas in each arm. Record the volume of gas in each tube. Test both gases with the splint test after inverting a test tube over each stopcock and removing about 15 ml of each gas into separate test tubes.

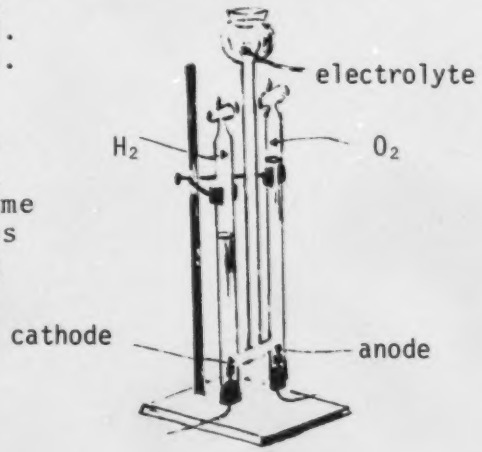


Figure D-14.

DATA

Observations:
Calcium + Water:
Calcium Oxide + Water
Sodium Aluminum Sulfate + Sodium Hydrogen Carbonate + Water:
Heating of Copper (II) Sulfate:

Name of Salt	Water Present?	Description of Residue
Sodium Carbonate		
Sodium Sulfate		
Potassium Sulfate		
Sodium Chloride		
Calcium Chloride		
Sodium Aluminum Sulfate		
Barium Chloride		
Iron (II) Sulfate		
Potassium Chloride		
Potassium Chlorate		
Cobalt (II) Chloride		
Nickel (II) Sulfate		

Name of Salt	Mass (initial)	Mass (final)	Gain or Loss	Description of Residue
Sodium Carbonate				
Sodium Sulfate				
Magnesium Chloride				
Potassium Nitrate				
Calcium Chloride				

Observations:
Sodium + Water
Potassium + Water
Heating of Phosphorus Litmus Test:

Electrolysis of Water:		
Name of Gas	Volume Produced	Splint Test

EXPERIMENT D-15: SOLUTIONS AND SUSPENSIONS

INTRODUCTION

The nature of the solution process is not completely understood by chemists. We shall assume in this experiment that in a solution there is an interaction between a substance that does the dissolving (solvent) and a substance that is dissolved (solute). This process probably involves chemical changes. After performing this experiment, you may be able to make a few helpful generalizations about solutions.

MATERIALS

Thermometer (°C); balance; corks; filter paper; test tubes; mortar and pestle; burner; small Erlenmeyer flask; funnel; test tube rack; test tube holder; graduated cylinder; starch; sugar; distilled water; copper sulfate ($CuSO_4$) crystals; sodium chloride (NaCl); potassium chromate ($KCrO_4$); calcium hydroxide ($Ca(OH)_2$); 0.01 M ammonia water; sodium thiosulfate ($Na_2S_2O_3$) crystals.

INSTRUCTIONS

Weigh out two 2.0 gram samples of copper sulfate crystals. Grind one sample to a powder in a mortar and pestle. Place both the ground and unground portions in test tubes about one-third full of water. Cork and shake both tubes. Record all observations and save these solutions for a later section of the experiment.

Select two small crystals of copper sulfate ($CuSO_4$) approximately equal in size and place each in a half-full test tube of water. Leave one tube undisturbed; cork the other and shake the contents. Does the crystal appear to be dissolving? How does agitation affect the rate of solution as you see it? Record all observations.

Place one-fourth of a teaspoonful of sugar in each of two test tubes. Fill one tube half full of *cold* water and the other tube half full of *hot* water. Cork and shake both tubes. Record your observations on the data sheet.

Examine the copper sulfate ($CuSO_4$) solutions saved from the first step. Do you observe any of the dissolved crystal settling out of the solution? Attempt to read some printed words through one of the tubes of the copper sulfate solution. Filter one of the tubes of solution with filter paper and a funnel. Pour the filtrate into a clean tube and compare its color with that of the unfiltered tube of solution. Do you notice any difference? Record all your observations.

Place about one-fourth teaspoonful of starch in a small flask partly filled with 50 ml of water. Cork the flask and shake vigorously. Attempt to read some printed words through this suspension. Set a test tube of the starch suspension aside until near the end of the laboratory period. At that time observe its clarity. Filter about 25 ml of the starch suspension and collect the filtrate in a clean tube. What observations can you record about this filtrate?

Fill a large test tube with 3.0 grams of powdered sodium chloride (NaCl). Add enough water to half fill the test tube and stopper it. Repeat the procedure using powdered potassium chromate ($KCrO_4$) and powdered calcium hydroxide ($Ca(OH)_2$). Shake each tube vigorously and allow to stand for about 2 minutes. Hold each tube up to the light and observe how much of each solute has dissolved. Then add 6.0 more grams of each solute to the proper test tube, stopper the three tubes, shake them vigorously, and allow them to stand for about 2 minutes. Observe the tubes to see if all the additional material dissolved in each test tube. Can you say that there is a limit to the amount of solid a solvent will dissolve? Record all your observations.

To 3 ml of water in a test tube add 2 or 3 crystals of sodium thiosulfate ($Na_2S_2O_3$) crystals. Shake and note the ease with which the solid dissolves at ordinary room temperature. To this solution add another small crystal of sodium thiosulfate. Shake to aid dissolving. Such a solution is said to be unsaturated. Save this solution after recording all of your observations.

To the above solution continue to add sodium thiosulfate ($Na_2S_2O_3$), two or three crystals at a time, until some remains undissolved after shaking for a while. When no more appears to dissolve in the solvent, allow the excess to settle, and pour off the liquid into a clean tube. Now drop a fresh crystal into this liquid. Observe the size of the crystal closely for a few minutes to see if it decreases in size. Record your observations on the data sheet.

Weigh out 15.0 grams of sodium thiosulfate ($Na_2S_2O_3$). Thoroughly clean a test tube before using. Rinse the tube several times with distilled water and then place 2 ml of distilled water into the tube. Dissolve the 15 grams of sodium thiosulfate in the 2 ml of

water, heating the tube as much as necessary. Do not let the solution boil over. When the solid is entirely dissolved, place a clean cork in the tube. Set the tube in a beaker of cold water carefully. After 10 or 15 minutes, examine the contents of the tube. This solution is a supersaturated solution. Remove the tube from the water and tap it sharply with your pen or pencil several times. If nothing happens, remove the cork and drop in a small crystal of the sodium thiosulfate. Feel the tube as soon as crystals begin to form. Use a thermometer to record any change in temperature. Record your observations on your data sheet.

Add about 5 ml of a 0.01 M ammonia water (ammonium hydroxide) solution to a test tube. Cautiously smell the contents of the tube. Heat the tube until half the volume remains again and smell the contents once more. Record all observations.

DATA

Observations:		
Copper Sulfate		
Ground	Unground	
Agitated	Undisturbed	
Cold	Hot	
Before Filtration	After Filtration	
Starch		
Before Filtration	After Filtration	
Various Solutes		
Sodium Chloride	Potassium Chromate	Calcium Hydroxide
Sodium Thiosulfate		
Unsaturated	Saturated	Supersaturated
Ammonia Water		
Before Heating	After First Heating	After Second Heating

EXPERIMENT D-16: A QUANTITATIVE STUDY OF THE EFFECT OF TEMPERATURE ON SOLUBILITY

INTRODUCTION

The amount of a pure substance that will dissolve in a solvent varies with the temperature of the solvent. In this experiment, we will determine the effect of heat upon the solubility of a salt in water. Each team in the laboratory will be assigned a certain temperature at which they will attempt to dissolve as much of the salt as possible. The data from all the teams will be collected and compiled into a graph from which predictions will be made.

MATERIALS

Evaporating dish; watch glass; large test tube; stirring rod; funnel; filter paper; 250 ml beaker; thermometer (°C); graduated cylinder; balance; wire guaze; ring and ringstand; clamp; burner; matches; solid potassium chloride (KCl); distilled water.

INSTRUCTIONS

Your instructor will assign your team a certain temperature for which you will determine the solubility of potassium chloride in water. Weigh a clean, dry evaporating dish with watch glass cover to the nearest 0.01 gram and set the dish aside for later use.

Fill a 250 ml beaker about two-thirds full of water and place it on a wire gauze supported by a ring stand over a Bunsen burner. Place about 15 ml of distilled water in a clean large test tube. Add 10.00 grams of potassium chloride (KCl) solid to the test tube of distilled water. Immerse the test tube in the beaker of water and clamp it to the ring stand. Heat the water until it reaches the temperature that you were assigned. Hold the water at this temperature for about 10 minutes. Stir every few minutes to insure saturation of the solution. Do not remove the stirring rod from solution.

Set up a funnel with filter paper for filtering the solution. Place the weighed evaporating dish below the funnel. Remove the test tube from the water bath. Take care to leave the solid undisturbed. Pour out the liquid into the funnel. Carefully weigh the evaporating dish and watch glass cover which contains the filtrate without any spilling. Evaporate the liquid to dryness by heating rapidly at first and then slowly. *Spattering indicates that you are heating the liquid too rapidly.* Allow the dish and cover to cool. Weigh again. Reheat. Cool and weigh once more. Repeat this process until two successive weighings agree to within ±0.05 grams. Proceed with the calculations and graphing of the data.

DATA

Observations:	
Weight of empty dish	g
Weight of evaporating dish and solution	g
Weight of solution	g
Weight of evaporating dish and dry KCl	g
Weight of KCl in solution	g
Weight of water in solution	g
Weight of KCl dissolved in 100 gram of water*	g

$$\text{*Weight of KCl dissolved in 100 grams of water} = \frac{\text{Weight of KCl in solution}}{\text{Weight of water in solution}}$$

DATA FROM OTHER TEAMS

Temperature at Which the Saturation took Place	Weight of KCl in 100 grams of Water
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	
9.	
10.	

Graph your results, using the horizontal axis for temperature and the vertical axis for the weight of KCl in 100 grams of water.

EXPERIMENT D-17: SOLUBILITY

INTRODUCTION

When a true solution is allowed to stand, the solute particles do not settle out on the bottom of the container. The solute particles are of molecular dimensions and will pass unchanged through filter paper. Substances in each of the three phases of matter may act as either solutes or solvents. In general we will be concerned

with solutions in which the solvent is a liquid. The solute may be a solid, liquid, or gas. The extent to which a solute dissolves in a solvent is known as its solubility.

MATERIALS

Test tubes; corks; timer; thermometer (°C); stirring rod; graduated cylinder; balance; sucrose; naphthalene; distilled water; benzene; iodine crystals; carbon tetrachloride; glycerine; potassium nitrate (KNO_3) crystals; ammonium chloride (NH_4Cl); sodium hydroxide (NaOH) pellets; concentrated sulfuric acid (H_2SO_4).

INSTRUCTIONS

Add 0.5 gram of ordinary sucrose to each of two test tubes and 0.5 gram of naphthalene to each of two other test tubes. Then add 5 ml of water to a test tube containing sugar and 5 ml of water to a test tube containing naphthalene. Shake each tube for 20 to 30 seconds and record in the data table the solubilities of each solute in water. Add 5 ml of benzene to the test tube containing the dry sucrose and 5 ml of benzene to the tube containing the dry naphthalene. Shake each tube for 20 to 30 seconds and record in the data table the solubilities of each solute in benzene.

Place a small crystal of iodine in the bottom of a test tube. Add 5 ml of water and 5 ml of carbon tetrachloride to the test tube. Shake vigorously. Record the solubility of iodine in carbon tetrachloride and in water. Base your conclusions on the intensity of the color of the iodine in the two solvents. Repeat the above using benzene and water. Record on the data sheet the extent to which water appears to dissolve in carbon tetrachloride and also in benzene.

Half-fill a small test tube with equal volumes of glycerine and water. Half-fill a second test tube with equal volumes of carbon tetrachloride and water. Stopper the two tubes, shake them vigorously, and allow them to stand for about 2 minutes. Observe whether or not solution has occurred and record observations on the data sheet. Pour an additional 3 ml of glycerine into the first test tube and the same quantity of carbon tetrachloride into the second test tube. Stopper the tubes and again shake them vigorously. Results?

Put 10 ml of water in each of three test tubes. Insert a thermometer into the water in the first test tube and record the temperature. To the water in the first test tube, add 5 grams of potassium nitrate (KNO_3). Gently stir the tube contents with the stirring rod *not the thermometer!* Note the temperature when all 5 grams have been dissolved, or almost completely dissolved. Repeat this procedure with the second test tube using 5 grams of ammonium chloride (NH_4Cl). To the third test tube add 5 pellets of sodium hydroxide (NaOH) trans-

ferring them from the bottle to the test tube by using a spatula (or wooden splint if spatulas are not available). *Caution: Sodium hydroxide is a powerful caustic hydroxide. Do not handle the pellets or permit them to touch your skin. Care in handling the solution should also be observed. If you should happen to come in contact with the sodium hydroxide, wash the area liberally with water and notify your teacher immediately.*

To 20 ml of water in a beaker *add carefully and with much stirring* 5 ml of concentrated sulfuric acid. Read the initial and final temperatures of the solution. *When working with concentrated acids always be aware of the location of the safety shower and the eyewash equipment.*

DATA

	Water	Benzene	Carbon Tetrachloride
1. Sugar			
2. Naphthalene			
3. Iodine			
4. Water			
5. Glycerine			

	Initial Temp. (°C)	Final Temp. (°C)
Potassium Nitrate + Water		
Ammonium Chloride + Water		
Sodium Hydroxide + Water		
Sulfuric Acid + Water		

EXPERIMENT D-18: A STUDY OF HARD WATER

INTRODUCTION

Calcium and magnesium compounds present in various quantities in water are responsible for a property known as "hardness." Hardness in water causes problems such as boiler scale in pipes and soap scum in basins. In this experiment we will test various types and samples of water to determine the degree of hardness that they possess.

MATERIALS

Two burets; 250 ml flasks with stoppers to fit; buret clamp; ringstand; ring; wire gauze; burner; matches; graduated cylinder; several beakers; watch glass; spatula; test tubes; funnel; filter paper; balances; timer; standard calcium chloride ($CaCl_2$) solution; soap solution; distilled water; tap water; rain water; solid calcium chloride ($CaCl_2$).

INSTRUCTIONS

Hardness is stated as a certain number of parts of calcium carbonate per million parts of water (p.p.m. $CaCO_3$). The soap-consuming power of a certain sample of water may be used to measure its hardness if a standard soap solution is available. The standardization of such a soap solution may be carried out in terms of a solution containing a known quantity of calcium ion.

Obtain from the supply two burets, 50 ml of a standard calcium chloride solution, 200 ml of a soap solution. Clean the burets. Rinse one with a few mls of the soap solution. Clasp it to the ring stand and fill it with the soap solution. Rinse and fill the other buret with the standard calcium chloride ($CaCl_2$) solution. Drain sufficient liquid from each to remove all air bubbles from the tip of the burets.

Run *exactly* 20 ml of the calcium chloride solution into a *clean* 250 ml Erlenmeyer flask anddilute with 30 ml of distilled water. Add 3 ml of the soap solution from the other buret, stopper the flask and shake it *vigorously*. Notice that the lather disappears rapidly. Continue the addition of soap solution until a lather is formed which covers the surface of the liquid and lasts for 3 minutes while the flask lies on its side. Record in the data table the buret reading for the amount of soap used. Repeat the titration.

In order that all determinations of hardness be made on the same sample, draw 300 to 400 ml of water from the tap and keep it in a stoppered bottle until needed. Keep the standarized soap solution in your drawer for use in all further "hardness" titrations.

Rinse the buret which contained the calcium chloride ($CaCl_2$) solution several times with water from the tap and then fill it with the tap water previously set aside. Add exactly 20 ml of it to the titration flask and dilute with 30 ml of distilled water. Determine the soap-consuming power of the sample of tap water by titrating with the standardized soap solution as before. The presence of magnesium compounds in the tap water may cause the appearance of a false end point some time before the true end point is reached. This false lather seldom lasts for three minutes. To be sure of making no mistake, read the buret after the titration is apparently finished and add 0.5 ml more of the soap solution. If the end point is a true one, the lather will increase in amount; but if false, the lather will disappear and the titration must be continued. Repeat the titration and record the data in the data sheet.

Place 100 ml of the tap water in a 250 ml beaker, cover with a watch glass, and heat to boiling. Boil for five minutes, cool to room temperature and dilute to the original volumes (100 ml) with distilled water. To a 20 ml sample of the boiled tap water add a 30 ml portion of distilled water. Titrate with soap solution and record your data. Repeat titration and record the data.

Add about 0.4 grams of solid $CaCl_2$ to 40 ml of distilled water in a clean beaker. Stir this solution until the solid is dissolved and thoroughly mixed in solution. This solution is known as "permanent" hard water. Divide the sample into two portions. Boil one sample for a few minutes and titrate it with the standardized solution. Test the unboiled sample in the same manner. Record all data.

If your instructor has collected some rain water recently, titrate a 20 ml sample (don't forget the 30 ml of distilled water) with the standard soap solution.

Now examine the discussion below the data table and perform all the required calculations to determine the hardness of all the samples you titrated.

DATA

	Standard CaCl		Tap Water		Boiled Tap Water		Permanent Hard Water		Rain Water	
	1	2	1	2	1	2	boiled	un-boiled	1	2
Sample size (ml)	20	20	20	20	20	20	20	20	20	20
Vol. of soap used (ml)										
Hardness (p.p.m.)	200	200								
Average hardness	200									

CALCULATIONS FOR SOAP STANDARDIZATION

Standard solution: 1 ml = .0002 grams of $CaCO_3$ (or 200 p.p.m.)

*Average** volume of soap used = _____

*in first tiration

$$200 \text{ p.p.m.} \times \frac{20 \text{ ml}}{\text{av. ml of soap}} = \text{_____ p.p.m. (equivalents of standard soap solution)}$$

HARDNESS CALCULATIONS

$$\text{Number of p.p.m. equiv. of standard soap solution} \times \frac{\text{ml. of soap used}}{20 \text{ ml}} = \text{p.p.m. of } CaCO_3 \text{ in sample}$$

EXPERIMENT D-19: COMPARISON OF VARIOUS CONSUMER WASHING PRODUCTS

INTRODUCTION

Soap is the common name for the sodium salts of fatty acids. The composition of the soap depends upon the nature of the fat which is used. Soap is made by treating a fat with an alkali. This type of reaction is called saponification. Modern detergents or cleansing agents are quite different from soap in chemical composition. In general, they are complex organic compounds, easily modified to meet specific needs. There are two general classifications of detergents: the sulfonated oils and sulfated long chain alcohols.

MATERIALS

Fat, lard, cottonseed, or olive oil; alcohol; 30% sodium hydroxide (NaOH) solution; distilled water; litmus paper; solid sodium chloride (NaCl); 0.5 M calcium chloride ($CaCl_2$); kerosene; scouring powder; wool yarns; lampblack or animal charcoal; marble chips; 1 M hydrochloric acid (HCl); clear limewater ($Ca(OH)_2$); trisodium phosphate (Na_3PO_4) solid; ammonium molybdate ($(NH_4)_2MoO_4$) solution; dilute ammonium hydroxide (NH_4OH) (1:2); solid sodium tetraborate ($Na_2B_4O_7$); tumeric paper; 1 M sodium hydroxide (NaOH); various soaps, detergents and water softeners; 250 ml flask; rubber stopper (one hole); glass tubing; burner; matches; evaporating dish; 250 ml beaker; glass plates; balance; graduated cylinder; timer; eye dropper; corks.

INSTRUCTIONS

Place about 50 ml of melted fat, lard, cottonseed oil or olive oil in a 250 ml Erlenmeyer flask. To this add 25 ml of ethyl alcohol and 15 ml of a 30% sodium hydroxide solution. *Caution: Sodium hydroxide is very caustic.* Place a one-hole rubber stopper fitted with a

long glass tube (60 cm) in the flask. The alcohol vapor will condense in this tube and run back into the flask. The alcohol speeds the reaction as it dissolves both the oil and the hydroxide. Boil the mixture *very gently* until the layers mix and start to solidify. Transfer the mixture to a large evaporating dish and heat until the alcohol is driven off. Constantly stir the mixture. Upon cooling, most of the soap will separate as a solid layer at the top. Then transfer the mixture to a large beaker and dissolve in as little boiling water as is necessary to liquefy it. Stir in 2 grams of solid sodium chloride. Then let it cool. When cold, lift out the cake of soap and wash it with cold water.

Prepare 2% solutions of the prepared soap, a commercial soap, and two different brands of detergents by dissolving 2.0 grams of each solid in 100 ml of distilled water. (Some of these solutions can be prepared and tested while the prepared soap is cooking.)

With a glass stirring rod, bring a drop of each of the solutions to various strips of red and blue litmus paper. Record whether they are acidic or basic on the data sheet.

Into four test tubes place 10 ml of distilled water. Add one drop of the solution, stopper the tube and shake it for a few seconds. Repeat until a good suds that lasts for three minutes is formed. Record the number of drops required. Repeat the suds-producing test with each of the soaps or detergent solutions and record all your data on the data sheet.

Fill four test tubes almost full with distilled water. Add two drops of the solution prepared earlier to the four tubes, so that you have dilute solutions of the prepared soap, the commercial soap, and two different detergents. Fill another tube just with distilled water to be used as a control. Cut five equal-length pieces of unwashed wool yarn (2 cm) from the supply. Roll each length into a small ball. Place the yarn balls on the surface of the tubes simultaneously. Observe if there is any difference in the sinking rate. Time the balls, indicating on the data sheet the length of time it took each ball to sink to the bottom.

Fill five test tubes half-full with distilled water. Add a few drops of kerosene to each tube. Then add 3-4 drops of the various solutions of soaps and detergents to the various different tubes. Stopper each tube and shake vigorously. Which soap or detergent emulsifies the kerosene the best? Which is the worst emulsifier? Record all of this on the data sheet.

Fill five test tubes half full with distilled water. Sprinkle only a *pinch* of lampblack or animal charcoal in the five tubes of water. Then add 3-4 drops of the various solutions of soaps and detergents to the different tubes of water. Stopper each tube and shake the

tubes vigorously. Which soap or detergent emulsifies the solid the best? Which is the worst emulsifier of the solid? Record all results on the data sheet.

Fill five test tubes halfway full with 0.5 M calcium chloride solution. Then add 3-4 drops of various solutions of soaps and detergents to the test tubes. Stopper each tube and shake vigorously. Which soap or detergent produces the best lather in this hard water? Which produces the least lather? Judge each and record the data.

Prepare 2% solutions of two water softeners by dissolving 2.0 grams of each solid in 100 ml of water.

Test for Carbonates: Into a test tube, fitted with a one-hole stopper and a delivery tube, place a piece of marble chip. Add 3 ml of 1 M hydrochloric acid. Pass the gas given off into 5 ml of clear limewater ($Ca(OH)_2$). The white precipitate formed is $CaCO_3$ and is the test for the presence of carbonate. Test each of the brands of water softener in the same way by placing 5 ml of the softener in the tube instead of the marble chip and then adding the acid. Trap any gas evolved with the limewater. Record all results.

Test for Phosphate: Dissolve 0.5 grams of trisodium phosphate (Na_3PO_4) in a half test tube full with distilled water. Add 10 ml of ammonium molybdate ($(NH_4)_2MoO_4$) solution. Warm gently, not to boiling. Add 1:2 ammonium hydroxide (NH_4OH) until it is basic to litmus. Add dilute (1:2) nitric acid (HNO_3) until the solution will turn blue litmus paper red. A yellow precipitate shows the presence of a phosphate. Now repeat this test with the several brands of water softeners by placing 5 ml of the brand being tested in the test tube and adding the 10 ml of ammonium molybdate solution to it. After completing the rest of the phosphate test, record your results on the data sheet.

Test for Borate: Dissolve 0.25 grams of sodium tetraborate ($Na_2B_4O_7$) in 5 ml of water. Add dilute (1:2) hydrochloric acid (HCl) until the effervescence stops and the solution turns blue litmus paper red. Moisten a piece of tumeric paper with the above solution. Dry it and if it is faint pink in color, a borate is present. Place one drop of 1 M sodium hydroxide (NaOH) on the paper. The formation of a greenish color proves the presence of a borate. Now test your samples of water softeners by placing 5 ml of the solution in the test tube instead of 0.25 grams of sodium tetraborate and proceed with the instructions above. Record your results on your data sheet.

Scouring powders usually consist of an abrasive and a water softener. The abrasive is usually silica, pumice, or feldspar. Thoroughly mix a teaspoonful of the scouring powder to be tested with 50 ml of

distilled water and filter after as much of the material has been dissolved as possible. Retain the filtrate and the residue.

Place some of the residue on a small plate of glass and rub it between two glass plates. Note whether it appears very gritty or not. Examine the glass plates for any evidence of scratching. Record your findings. Acidify about 5 ml of the filtrate with nitric aicd and apply the phosphate test described above. Acidify another portion of the filtrate with hydrochloric acid and test for the presence of borate. Apply the carbonate test to a dry portion of the scouring powder. Record the results of the phosphate, borate, and carbonate test on your sheet.

DATA

	Prepared Soap	Commercial Soap Brand:	Detergent #1 Brand:	Detergent #2 Brand:
Acid-Base Test				
Suds-Producing Test				
Wool Yarn Test				
Kerosene Emulsifying Test				
Charcoal Emulsifying Test				
Hard Water Lather Test				

	Water Softener #1 Brand:	Water Softener #2 Brand:	Scouring Powder Brand:
Carbonate Test			
Phosphate Test			
Borate Test			
Abrasion Test			

EXPERIMENT D-20: TESTING WATER SAMPLES FOR DISSOLVED OXYGEN

INTRODUCTION

Water is a good solvent for many gases. Water can dissolve over a thousand times its own volume of ammonia gas. Oxygen and nitrogen, on the other hand, are only very slightly soluble in water. Otherwise there would be none of these gases in the atmosphere, all having dissolved in the seas and oceans. The slight solubility of oxygen in water, however, is most important to aquatic animals for they depend on this for respiration.

MATERIALS

Test tubes; glass tubing; burner; stirring rod; sample bottles; graduated cylinder; 50 ml pipet; Erlenmeyer flasks; buret; buret clamp; tweezers; solid potassium hydroxide pellets; solid sodium pyrogallate; various water samples; concentrated sulfuric acid; 0.025 N sodium thiosulfate solution; 3.2 M manganous sulfate solution; starch solution; alkaline-iodide solution.

INSTRUCTIONS

Qualitative Test: Completely fill a large test tube with the sample of water to be tested. Using tweezers, drop three tablets of potassium hydroxide (KOH) into the solution. Rubber stopper the test tube. Seal shut a 2.5 cm length of glass tubing by heating one end of it in the burner flame until the ends fuse together. Cool the glass tubing and then place some solid sodium pyrogallate in the tube. Pack the sodium pyrogallate down into the fused end of the tube by using a thin glass stirring rod. Cover the exposed surface of the solid with a small amount of water from the sample being tested. Carefully drop the tube with the sodium pyrogallate in it into the test tube with the water sample and replace the rubber stopper into the end of the test tube. Be certain that the test tube is filled to the brim to minimize air spaces that will affect the validity of the results. Repeatedly invert and reinvert the test tube. The liquid will develop a brownish tint (an intermediately purple tint may appear but may be disregarded). The intensity of the brownish tint will vary with the oxygen content--the deeper the color the greater the amount of dissolved oxygen. Save all the tubes from the tests of the various water samples in order to make comparisons.

Quantitative Test: Fill a 250 ml Erlenmeyer flask with 100 ml of the water sample to be tested (two 50 ml pipet portions). This water has been treated with manganous sulfate, alkaline-iodide solution, and sulfuric acid in order to preserve it so that you can test it. At this point, the oxygen equivalent in the sample

is represented as free iodine. Set the flask against a white background. Fill a buret with 0.025 N sodium thiosulfate soltuion. Record the buret initial reading on the data sheet.

If the water sample is orange in color, rapidly add some thiosulfate solution to the sample until it becomes pale and straw colored. Then add 10 ml of starch indicator solution. A blue color should develop. Continue titrating while swirling and sample cautiously until the blue color disappears. Stop at this point and ignore any return of color. Record the final buret reading and subtract that value from the inital reading to give the amount of thiosulfate used. Repeat this titration with the various water samples that are to be tested. Record all the data on the data sheet.

DATA

Identification of Water Samples

Sample #1 _____ Sample #4 _____

Sample #2 _____ Sample #5 _____

Sample #3 _____ Sample #6 _____

Quantitative Test for Oxygen

	Samples					
	#1	#2	#3	#4	#5	#6
Initial Buret Reading						
Final Buret Reading						
Amount of Sodium Thiosulfate Used						

Rank order of water samples (from the highest content of dissolved oxygen to the lowest content)

1st_____ 2nd_____ 3rd_____ 4th_____ 5th_____ 6th_____

EXPERIMENT D-21: A STUDY OF SOME IMPURITIES IN WATER SAMPLES

INTRODUCTION

The contaminants that occur naturally and unnaturally in water supplies can be detected, and in many cases removed. In this experiment, we will be analyzing various water samples for the presence of suspended solids, mercury, lead, nitrates, sulfates, calcium, chlorides, phosphates, and other organic by-products of sewage decomposition. Some of these contaminants are directly "dumped" into the water resources. Others are absorbed by the rain as it falls through our polluted air. And still others reach the water supply by being adsorbed by the rain water runoff which eventually ends up in underground waters and then lakes and streams.

MATERIALS

Nessler's reagent; tin (II) chloride ($SnCl_2$) solution; aniline; solid iron (II) sulfate ($Fe(SO_4)_2$) ; concentrated sulfuric aicd (H_2SO_4); saturated silver sulfate (Ag_2SO_4) solution; 3 M sodium hydroxide (NaOH); saturated bromine water; benzidine solution; 6 M ammonium hydroxide (NH_4OH); 0.1 M potassium permanganate ($KMnO_4$); concentrated nitric acid (HNO_3); 1 M silver nitrate ($AgNO_3$); 0.25 M barium chloride ($BaCl_2$) solution; concentrated ammonium oxalate ($(NH_4)_2C_2O_4$) solution; 1 M acetic acid ($HC_2H_3O_2$); 0.5 M ammonium molybdate ($(NH_4)_2MoO_4$) solution; filter paper; eyedroppers; 150 ml and 250 ml beakers; watch glass; spatula; funnel; balances; graduated cylinder (10 ml); supply of water samples; ringstand; ring; wire gauze; burner; matches.

INSTRUCTIONS

Dissolved Solids: Obtain several watch glasses. Weigh each clean watch glass after properly labeling them with reference to the water sample supply. Include one watch glass for distilled water. Place 2 ml or 40 drops of each water sample and distilled water on the watch glasses. Evaporate the samples over water baths. When cool, weigh each watch glass to determine the mass of the solid residue remaining after evaporation. Examine each residue with a hand lens and record all observations on the data sheet.

Test for Detecting Mercury: Put two drops of the test solution on a strip of filter paper. Then add two drops of a *fresh* tin (II) chloride ($SnCl_2$) solution. Finally add two drops of aniline. A black to brown color on the paper indicates the presence of mercury. Record your results.

Test for Detecting Lead: Place two drops of the solution to be tested on a strip of filter paper. Then place in order, two drops of 3 M sodium hydroxide (NaOH), two drops of saturated bromine water, and then four drops of the 6 M ammonium hydroxide solution (NH_4OH) on the test solution. Allow the drops to evaporate and dry. Then add

drops of the benzidine until a reaction occurs. The intensity of the blue color is directly related to the amount of lead present in the water sample.

Test for Detecting Ammonia: The presence of ammonia in water samples usually indicates contamination by organic matter. To about one-third of a test tube full of water sample, add several drops of Nessler's reagent. Observe all changes. If little reaction is evident, heat gently.

Test for Detecting Organic Matter: To a third full test tube of water sample add enough 0.1 M potassium permanganate ($KMnO_4$) solution drop by drop to color the water a distinct violet tinge. Divide this solution into two equal portions in two test tubes. To one of the tubes add about 2 ml of concentrated sulfuric acid (H_2SO_4). Now heat both test tubes. The tube to which no acid was added is a control which will aid you in checking the change in color by comparison. If the color change is not distinct, let both tubes stand until the close of the period and again examine and compare them. Record all observations in color change on the data table.

Test for Detecting Calcium: To about one-third of a test tube of water sample, add 1 ml of acetic acid ($HC_2H_3O_2$) and 3 ml of ammonium oxalate ($(NH_4)_2C_2O_4$) solution. If no change is immediately evident, let the tube stand for 10 minutes; then compare its appearance with that of a tube of clear water. Record all observations.

Test for Sulfate Ion: Sulfates in water usually indicate some hardness. To a third test tube full of the water to be tested add 1 ml of concentrated nitric acid (HNO_3) and to this slowly add about 5 ml of 0.25 M barium chloride ($BaCl_2$) solution. Describe the results on your data sheet.

Test for Chloride Ion: To a test tube one-third full of test solution add 1 ml of concentrated nitric acid (HNO_3); next add a few drops of 1 M silver nitrate ($AgNO_3$) solution. To confirm the presence of the chloride ion add 2 ml of 6 M ammonium (NH_4OH) solution. If the precipitate or cloudiness in the water sample disappears, it was the silver chloride and is a positive result, to be recorded in the data table.

Test for Detecting Nitrate Ion: Many other ions interfere with the test for the presence of nitrate ion so they must be removed if they are present. They can be removed by adding saturated silver sulfate solution to the sample by drops until nothing happens. Then filter the residue from the solution. After removing these interfering ions put a small crystal of ferrous sulfate – iron (II) sulfate ($FeSO_4$) – on a watch glass. Place a drop of the filtered test solution on the crystal. Let a drop or two of concentrated sulfuric acid (H_2SO_4)

run into the solution-crystal mixture from the side by allowing the drops to roll down the watch glass into the liquid. Observe whether a brown ring or line is present. Place the watch glass carefully on a piece of white paper to check. The presence of a brown ring or line is a positive test for the nitrate ion.

Test for Detecting Phosphate Ion: Place a drop or two of the water sample test solution on a piece of filter paper. Then add two drops of ammonium molybdate ($(NH_4)_2MoO_4$) solution followed by two drops of benzidine solution. Hold the paper over a beaker of 6 M ammonium hydroxide (NH_4OH). When most of the acid (phosphomolybdic acid) has been neutralized, a blue color will form: the more intense the color, the more phosphate ion present in the water sample being tested.

All of the above tests should be run on all the water samples available, tap water, and distilled water.

DATA

Samples	Location	Observations
#1		
#2		
#3		

Evaporation Residues		
	Mass of Residue	Observations
Sample #1		
Sample #2		
Sample #3		
Tap Water		
Distilled Water		

Analysis for Various Ions and Molecules						
	Sample #1		Sample #2		Sample #3	
	Present	Obser.	Present	Obser.	Present	Obser.
Mercury						
Lead						
Ammonia						
Organic Matter						
Calcium						
Sulfate						
Chloride						
Nitrate						
Phosphate						

EXPERIMENT D-22: A STUDY OF pH, INDICATORS, AND ALKALINITY OF WATER SAMPLES

INTRODUCTION

The relative strength of an acid or a base is determined by the degree of ion formation of the acid or base in solution. An exact way to describe the ion formation of an acid or a base is to indicate the concentration of hydrogen ions in the solution. This is called the pH of the solution. The pH can range from 1.0 for a strong acid to 14.0 for a strong base. A solution with pH of 7.0 is neutral, neither acid or base. An indicator is a substance whose color in solution depends upon the hydrogen ion concentration, the pH. Most indicators are dyes that change color over a very narrow pH range. In this experiment we will study a series of standard solutions of known hydrogen ion concentration (pH). These standard solutions will then be used to determine the pH of some unknown substances.

The alkalinity of a water sample is the capacity of that water sample to accept hydrogen ions. Alkalinity is usually imparted by the bicarbonate (HCO_3^-), carbonate (CO_3^{-2}), and hydroxide (OH^-) components of a natural or treated water supply. The alkalinity will be determined in this experiment by titration with a standard acid solution with the endpoint indicated by means of the color change in a dye, an indicator.

MATERIALS

125 ml Erlenmeyer flask; 50 ml beaker; eyedropper; buret; 50 ml pipet; white paper; small test tubes; 0.5 ml pipet; china marker; red and blue litmus; graduated cylinder; 0.1 M hydrochloric acid (HCl); 0.1 M sodium hydroxide (NaOH); indicator solutions: congo red, methyl violet, alizarin yellow, indigo carmine, phenolphthalein, methyl orange, mixed bromcresolgreen-methyl red; standard 0.02 N sulfuric acid (H_2SO_4), 0.1 N sodium thiosulfate ($Na_2S_2O_3$) solution.

INSTRUCTIONS

Obtain five test tubes, clean them thoroughly and rinse them with distilled water. Label the test tubes with numbers 1 through 5. Place 5 ml of 0.1 M HCl in test tube 1. With a pipet, remove 0.5 ml from test tube 1 and transfer the 0.5 ml to test tube 2. Add 4.5 ml of distilled water to test tube 2. Mix well by rotating the test tube. Remove 0.5 ml of solution from test tube 2 and transfer it to test tube 3. Add 4.5 ml of distilled water to test tube 3 and mix. Continue this procedure of transferring 0.5 ml from the preceeding test tube into the next and adding 4.5 ml of distilled water until you have filled the 5 test tubes. What you now have are five HCl solutions with the following concentrations: 0.1 M, 0.01 M, 0.001 M, 0.0001 M, and 0.00001 M. These concentrations correspond to pH's of 1, 2, 3, 4, and 5 respectively. Place a drop of methyl violet indicator in each test tube. Observe the color in each tube and record all observations. Save the test tubes for comparisons later in the experiment.

Obtain five more test tubes and follow the steps outlined in the previous paragraph to prepare the dilutions of 0.1 M hydrochloric acid. After the dilutions are completed add a drop of congo red indicator to each test tube. Observe the color range of the solutions and record your observations. Save these tubes also.

Obtain 5 ml of 0.1 M sodium hydroxide solution and prepare *two* sets of dilutions using the same procedure as indicated in the first paragraph. Place one drop of alizarin yellow indicator in one set of the dilutions and one drop of indigo carmine indicator in the other set of dilutions. Record all observations on the data sheet and save the sets of dilutions.

Obtain 10 ml of an unknown sample. Pour 5 ml of the unknown into each of two test tubes. Determine if the unknown is acid or base by using litmus paper. If the solution is acidic, add methyl violet to one tube and congo red to the other. If the solution is basic, add alizarin yellow to one tube and indigo carmine to the other. Compare the two tubes with the color standards you prepared above. From the comparison and your knowledge of the concentration of the standards, determine the concentration of the unknown and record it on your data sheet.

Choose a water sample which you wish to test for its alkalinity. Pipet a sample of the water to be tested (50 ml) into a 125 ml Erlenmeyer flask. Remove any free residual chlorine in the sample by adding 1 drop of 0.1 N sodium thiosulfate solution. Then add 2 drops of phenophthalein indicator solution to the sample. Titrate the solution with the standard acid solution (0.02 N sulfuric acid) by placing the standard acid in a buret and putting a sheet of white paper under the Erlenmeyer flask. Do not forget to swirl the flask as you are adding the acid, but not too vigorously. At the endpoint the pink color will disappear. Perhaps it will only take a few drops to achieve this at the endpoint so be careful when titrating not to overshoot the endpoint.

After you have reached the endpoint of the above titration add 3 drops of the mixed indicator bromcresol green-methyl red to the solution in which you just titrated the phenolphthalein endpoint. Again titrate with the standard acid over a white surface. You should expect the following color changes: greenish blue to light blue with lavender gray, to light pink gray with bluish tinge to light pink. At the light pink color the titration is complete. Record the buret readings again on your data table for both trials. Repeat the entire procedure with another water sample so that you will have two trials of this titration. Record your buret readings on the data sheet.

To check your titrations above, try to titrate a water sample using only one indicator, methyl orange. Add 2 drops of this indicator to 50 ml of the water sample and titrate with standard acid over a white surface until the indicator turns from orange to pink. Record you data on your data table.

DATA

Standard Test Tube Colors						
		0.1 M	0.01 M	0.001 M	0.0001 M	0.00001 M
Acidic Indicators	Methyl Violet					
	Congo Red					
Basic Indicators	Alizarin Yellow					
	Indigo Carmine					

Unknown Analysis				
Acid	Base	Concentration	Indicator Used Name	Color
1.				
2.				

Titration of Water Sample						
	Phenolphthalein		Bromcresol green - Methyl red		Methyl Orange	
	Trial 1	Trial 2	Trial 1	Trial 2	Trial 1	Trial 2
Final Buret Reading						
Initial Buret Reading						
Total Volume of Acid Used						

Water Sample Identification Data	
Source	
Date of Removal	
Description of Location	

EXPERIMENT D-23: WATER TREATMENT AND SURFACE CHEMISTRY

INTRODUCTION

The study of adsorption and the colloidal state of matter can be grouped under the heading of surface chemistry. The colloidal state is a fundamental state of matter. In this experiment we will investigate how the principles of adsorption and colloidal chemistry are used in the treatment of water to remove dye pollutants.

MATERIALS

Kaolin; methyl violet; methylene blue; malachite green; alizarin; aluminum hydroxide ($Al(OH)_3$); boneblack; molasses solution; cider vinegar; hydrogen sulfide (H_2S) solution; 6 M ammonium hydroxide (NH_4OH); mineral oil; soap solution (concentrated); 0.02 M $CaCl_2$; alcohol; red litmus paper; 125 ml Erlenmeyer flask; stopper to fit flask; balance; distilled water; graduated cylinder; two 250 ml beakers; burners; matches; crucible and cover; eyedropper; funnel; filter paper; timer; stirring rod; large test tubes.

INSTRUCTIONS

Place kaolin, about 2 cm in depth, into a 125 ml Erlenmeyer flask. Fill it with 75 ml of distilled water and stopper. Then add three drops of methyl violet solution, shake the flask for a minute, then allow it to stand. What observations can you record about the dye and its adsorption? Add more drops of the methyl violet solution. Has it been adsorbed? Try to add more drops of the solution and see if it is adsorbed. Record the numbers of drops adsorbed and all other observations on the data sheet. Remove some of the kaolin and treat it with boiling water to see if any of the dye can be extracted from the kaolin. Also try to extract the adsorbed dye with an alcohol solution. Repeat this procedure with the other dyes available--methylene blue, malachite green, alizarin, etc. Record all observations about the selective adsorption of dyes by kaolin.

Dissolve 10 grams of aluminum hydroxide ($Al(OH)_3$) in 100 ml of distilled water. Divide the solution and place halves into two 250 ml beakers. Add 6 M ammonium hydroxide (NH_4OH) to each solution until litmus paper indicates that the solutions are basic (red turns to blue). Then add enough water to the beakers so that they are three-fourths full. To one beaker add 20 ml of the alizarin solution. To the other beaker add 20 ml of a solution of methylene blue. Boil for a minute then allow the precipitate to settle. Pour off the solutions and examine the precipitate to see if any dye was adsorbed by it. Wash the precipitates with 50 ml of distilled water, allowing them to settle. Observe whether any dye was removed by the water from the solid. Wash the precipitates again after pour off the water with 50 ml of acetic acid. Does this solution remove the dyes? Record all observations on your data sheet.

Dilute 5 ml of the methyl violet solution to 100 ml with distilled water and place half of the solution in a 125 ml Erlenmeyer flask. Reserve the remainder for comparison. Add a heaped teaspoonful of crushed wood-charcoal to the solution and shake for a few minutes. After shaking with the dye, filter or allow the charcoal to settle. Compare the tint of the solution above the charcoal with the original solution and record your observations. Wash the charcoal with distilled water after pouring off the liquid. Shake it well, allow it to settle, then observe the tint of the liquid above the charcoal with 10-15 ml of alcohol; and observe the tint of the solution.

Activate about 2 teaspoonfuls of boneblack (animal charcoal), by heating it for 5 minutes in a covered crucible. Place a third of this activated charcoal in a test tube with 10 ml of a molasses solution. Shake well and heat to boiling. Filter the mixture and examine the filtrate. Record your observations on the data sheet. Repeat the procedure using cider vinegar in pace of the molasses solution and observe what happens.

Mix the remaining activated charcoal with 10 ml of a hydrogen sulfide solution. Shake the mixture vigorously at 2 minute intervals for about 15 minutes. Record any changes in odor of the filtrate after filtering it.

DATA

Adsorption of Dyes of Kaolin		
Name of Dye	No. of Drops of Dye Solution Adsorbed	Observations
Methyl Violet		
Methylene Blue		
Methyl Orange		
Malachite Green		
Indigo Carmine		
Congo Red		
Picric Acid		
Eosine		

Adsorption of Dyes by Aluminum Hydroxide	
Name of Dye	Observations
Alizarin	
Methylene Blue	

Adsorption of Color by Wood Charcoal Observations

Adsorption of Odors by Activated Animal Charcoal Observations		
Molasses Solution	Cider Vinegar	Hydrogen Sulfide Solution

EXPERIMENT D-24.1A: THE STUDY AND IDENTIFICATION OF CERTAIN METALLIC IONS (PART I)

INTRODUCTION

Chemists have divided the elements into two large groups: the metals and the nonmetals. All metals have similar properties but they differ in chemical reactivity. One of the factors which influences chemical reactivity is the energy absorbed when metallic atoms lose electrons to become positively charged ions. Metals also form salts with nonmetals and these salts have varying degrees of solubility in water. In this experiment you will be testing and identifying the metallic ions in solid crystals and water solutions of these ions.

MATERIALS

Blowpipe; platinum or nichrome wire (glass mounted); charcoal blocks; Bunsen burner; cobalt glass plate; crucible (porcelain); funnel; filter paper; graduated cylinder; powdered borax ($Na_2B_4O_7$); salts of Ni, Co, Cr, Mn, Cu, Fe(III), Al, Mg, and Zn; 3 M ammonium hydroxide (NH_4OH); 0.02 M cobalt nitrate ($Co(NO_3)_2$); concentrated hydrochloric acid (HCl) (12 M); solutions (0.01 M) of barium chloride ($BaCl_2$), calcium chloride ($CaCl_2$), lithium chloride (LiCl), potassium chloride (KCl), sodium chloride (NaCl) strontium chloride ($SrCl_2$); saturated copper (II) chloride ($CuCl_2$).

INSTRUCTIONS

Borax Bead Tests: Bend the end of a platinum or nichrome wire which is attached to a glass rod into a loop about 3 mm in diameter. Heat the loop red hot and dip it at once into powdered borax. Heat again in the outer region of the bunsen burner flame. The borax first swells as it loses its water of crystallization, then melts to a clear glassy bead. Repeat this procedure until a bead about the size of a drop of water has been formed. Dip this bead while still hot into a solution of a little of the salt to be tested. Heat again in the outer region of the flame until the bead has melted; then allow the bead to cool. If color is not evident in the bead, repeat. Carefully examine the bead in a good light, noting the color. To remove the bead from the wire, heat it until melted and immediately shake it off into a waste container. Make a fresh bead and test the other metals as indicated on the data sheet. Record all of your observations on the data table.

Cobalt Nitrate Tests: Close the openings of the Bunsen burner to produce a luminous flame about 4 cm high. Hold the tip of the blowpipe just inside the flame about 2 cm above the burner top and blow the flame down at an angle on the substance to be heated. To oxidize a substance, hold it in the outer tip of the blowpipe flame. To heat the substance in a reducing region, hold the substance nearer the burner and blow the inner portion of the flame upon the substance. Make a shallow cavity in a charcoal block. Put a little solid zinc salt in the cavity and heat it strongly in the oxidizing flame. If the salt tends to blow away, moisten with a drop of water. Cool and note the color. Add a drop or two of dilute cobalt nitrate ($Co(NO_3)_2$) solution and again heat strongly. Cool and note the characteristic color. Record all results in the data table. Clean out the cavity by scraping it, or make a new cavity, and test an aluminum salt in the same manner. Repeat the test for a magnesium salt in the same manner. The magnesium test is very faint so avoid using too much cobalt nitrate and heat as strongly as possible. Examine it near a window in daylight to ascertain its color.

Flame Tests: The platinum wire should always be cleaned before and after using. The wire, when clean, will not color the flame. To clean the wire alternately heat it and dip it in a little concentrated hydrochloric acid (HCl) until no color is produced in the flame. Do not heat the wire too close to the glass handle as the glass may crack. Discard the acid when through using it. Dip the clean platinum wire in a solution of the salt to be tested. Hold the wire with the solution on it in the lower part of the outer blue flame of the burner. Carefully observe any flashes of color as some colors do not remain long. Make flame tests using salts of the metals indicated in the data table. When observing the color of sodium flame, use a piece of cobalt glass. If the color persists, use two or three thicknesses of glass. Observe the color of a potassium flame through the cobalt glass. Now observe the flame of a mixture of sodium and potassium salts with the naked eye. Then observe the flame of the mixture through cobalt glass. Record all your observations on the data sheet. Be very specific about the intensity and color of the various flames observed.

DATA

Borax Bead Test		
Metal Ion	Formula of Compound Tested	Color of Base
Nickel		
Cobalt		
Chromium		
Manganese		
Copper		
Iron		

Cobalt Nitrate Test			
Metal Ion	Formula of Compound Tested	Color After First Heating	Color After Second Heating
Zinc			
Aluminum			
Magnesium			

Flame Test		
Metal Ion	Formula of Salt Used	Color of Flame
Lithium		
Sodium		
Potassium		
Calcium		
Barium		
Strontium		
Copper		

EXPERIMENT D-24.2A: THE STUDY AND IDENTIFICATION OF CERTAIN METALLIC IONS (PART II)

MATERIALS

Filter paper; 3 M hydrochloric acid (HCl); 3 M nitric acid (HNO_3); 0.5 M lead nitrate ($Pb(NO_3)_2$); 0.5 M mercury (I) nitrate ($HgNO_3$); 0.1 M silver nitrate ($AgNO_3$); 0.5 M potassium chromate (K_2CrO_4); 1 M magnesium chloride ($MgCl_2$); concentrated ammonium hydroxide (NH_4OH); litmus paper (blue and red); dolomite (calcium magnesium carbonate); distilled water; solid ammonium chloride (NH_4Cl); 1 M ammonium carbonate ($(NH_4)_2CO_3$); 1 M sodium hydroxide (NaOH); 0.5M mercury (II) nitrate ($Hg(NO_3)_2$); 1 M potassium iodide (KI); 1 M tin (II) chloride ($SnCl_2$); 3 M ammonium hydroxide (NH_4OH); 1 M ammonium chloride (NH_4Cl); balances; stirring rod.

INSTRUCTIONS

Test for Magnesium: To 5 ml of a solution of a magnesium salt add an equal volume of ammonium chloride solution. Make the solution strongly basic with concentrated ammonium hydroxide and add 1 ml of disodium phosphate (Na_2HPO_4). The precipitate formed is $MgNH_4PO_4$ and this reaction is used as a test for magnesium. Filter the precipitate and transfer it to a porcelain crucible and heat it strongly. Test the escaping vapor with moist litmus paper.

Weigh out 0.5 grams of dolomite and place it in 5 ml of dilute hydrochloric acid and heat to complete the reaction. Add water to dilute the solution to 20 ml. Then filter the solution. Dissolve a gram of ammonium chloride solid in the filtrate, make it distinctly basic with ammonium hydroxide (test with litmus paper to be sure) and then add about 10 ml of ammonium carbonate solution. Filter and add a solution of disodium phosphate (Na_2HPO_4) to the filtrate. This procedure is the analytical method employed for the separation of Mg^{+2} from Ca^{+2}. The calcium is precipitated as $CaCO_3$ and the magnesium as $MgNH_4PO_4$.

Identifying Lead, Mercury (I), and Silver Ions: In separate test tubes place 2 ml portions of the solutions of lead, mercury (I), and silver salts (nitrates). To each solution add dilute hydrochloric acid until no further reaction occurs. Add the acid a few drops at a time, shake well and allow to settle after each addition. Heat the contents of each tube just to boiling. To each precipitate add a few drops of ammonium hydroxide.

In separate test tubes obtain 3 ml of the solutions of lead, mercury (I), and silver salts. Place the solutions into the *same* beaker. To the mixture of solutions add dilute hydrochloric acid (3 M) until precipitation is complete. Filter the precipitate and wash it with a little cold water to remove any excess acid. Treat the residue on the filter paper with 5 to 10 ml of *boiling* water, catching the filtrate in a clean beaker. Add a few drops of potassium chromate solution to the filtrate fails to react with the potassium chromate solution. Record observations. Now treat the residue with about 5 ml of dilute ammonium hydroxide, catching the filtrate (silver ions), in a clean beaker. To this second filtrate add dilute nitric acid (3 M) until the ammonium hydroxide is completely neutralized (as indicated by litmus paper). Record your observations in the data table.

Comparisons Tests for Mercury (I) and (II): Add some hydrochloric acid to a solution of mercury (I) nitrate. Allow the precipitate to settle, pour off most of the liquid, and add ammonium hydroxide. This is a test for mercury (I) salts. To a small portion of mercury (I) nitrate solution add sodium hydroxide solution; to another

portion add ammonium hydroxide. Describe the results on the data sheet. Repeat all of the above instructions but use a solution of mercury (II) salt. Compare the solubility of the two solutions and record all observations.

DATA

Metal Ion	Observations of Characteristic Reaction
Magnesium	
Calcium	
Lead	
Mercury (I)	
Silver	
Mercury (II)	

EXPERIMENT E-1: THE PREPARATION OF SOME ORGANIC COMPOUNDS

INTRODUCTION

Organic compounds are compounds which contain carbon atoms. Organic compounds are classified according to the kinds of atoms in the compound and kinds of bonds formed between them. For example, the hydrocarbons are organic molecules containing only hydrogen and carbon atoms. The alkane series of organic compounds are hydrocarbons containing only single bonds between pairs of atoms. Most organic compounds are considered to be derivatives of the hydrocarbon series of compounds. The characteristic properties of organic compounds other than hydrocarbons are due mainly to a functional group which has replaced one of the hydrogen atoms of a hydrocarbon molecule. For example, in an alcohol, an OH group has replaced one of the hydrogen atoms. The purpose of this experiment is to survey some of the properties and characteristics of the various kinds of hydrocarbons and their derivatives.

MATERIALS

Pneumatic trough; test tubes; 250 ml beaker; ringstand; ring; burner; matches; test tube clamp; rubber tubing; one-hole stopper; glass tubing; splints; litmus paper; copper wire; soda lime solid; calcium carbide (CaC_2) solid; sodium acetate ($NaC_2H_3O_2$) solid; 3 M sulfuric acid (H_2SO_4); ethyl alcohol; methyl alcohol.

INSTRUCTIONS

Place 1.0 gram of sodium acetate solid and 2.0 grams of soda lime in a large test tube and mix thoroughly. Heat the mixture and collect the gas given off by displacement of water (see Figure E-1.1). Collect enough gas to fill two test tubes. Observe the color, odor and solubility of the methane gas in the first test tube. Bring a burning splint to the mouth of the second test tube of gas. Record all of your observations on the data sheet.

Figure E-1.1.

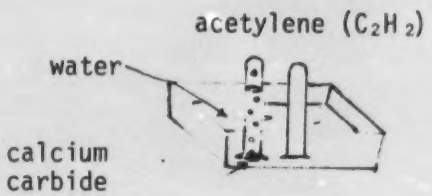


Figure E-1.2.

Fill two test tubes with water and invert them in the pneumatic trough used in the above preparation (see Figure E-1.2). Place a piece of calcium carbide, CaC_2, with forceps under the test tubes and collect two test tubes of the gas, acetylene. Use one of the test tubes to note the color, odor and solubility of the actylene gas. Bring a burning splint to the mouth of the second test tube of gas. Record your observations.

Place 1.0 gram of sodium acetate ($NaC_2H_2O_2$) in a large test tube. Add 2 ml of 3 M sulfuric acid (H_2SO_4). Warm the solution gently and carefully over a Bunsen burner and cautiously note the odor. Describe the odor on the data sheet. Place some moistened blue litmus paper in the vapor. Record your observations of acetic acid, commonly known as vinegar.

To the acid prepared in the above paragraph add 2 ml of ethyl alcohol (C_2H_5OH). Warm the solution gently and carefully in a beaker of boiling water for 2 to 3 minutes. Pour the contents of the test tube into a smaller beaker. Note all the physical characteristics of the substance formed which is an ester called ethyl acetate. Record all these observations on your data sheet.

Pour 1 ml of methyl alcohol (CH_3OH) into a small test tube. Make a spiral 25-30 cm long of copper wire by wrapping the wire around a pencil. Fasten one end of the spiral to a handle made from a pencil. Heat this coil of copper over a Bunsen burner until it is red hot, then quickly plunge it into the methyl alcohol. Reheat the wire and repeat two or three times until you can detect a distinct odor. This odor belongs to another type of organic molecule called an aldehyde, specifically formaldehyde. Record all your observations of this compound on the data table.

DATA

Preparation of Methane (alkane)--A Saturated Hydrocarbon			
Odor	Color	Solubility	Combustibility

Preparation of Acetylene (alkyne)--An Unsaturated Hydrocarbon			
Odor	Color	Solubility	Combustibility
Equation: Calcium Carbide + Water → Actylene↑ + Calcium Oxide CaC_2 + _____ → C_2H_2↑ + _____			

Preparation of an Organic Acid--Acetic Acid		
Odor	Color	Litmus Test

Equation: Sodium Acetate + Sulfuric Acid → Acetic Acid + Sodium Sulfate

$NaC_2H_3O_2$ + _____ → $HC_2H_3O_2$ + _____

(don't forget to balance it!)

Preparation of an Ester--Ethyl Acetate	
Odor	Color

Equation: Acetic Acid + Ethyl Alcohol → Ethyl Acetate + Water

_____ + C_2H_5OH → $CH_3COOC_2H_5$ + _____

Preparation of an Aldehyde--Formaldehyde
Odor

EXPERIMENT E-2: QUALITATIVE TESTS FOR SOME ORGANIC MOLECULES IN COMMON FOODS

INTRODUCTION

In all the different kinds of food which are available to us, there are only three basic kinds of nutrients--carbohydrates, fats, and proteins--plus minerals and, of course, water. Our bodies need fairly definite amounts of each of these each day. The carbohydrates and fats are needed to provide energy for our daily activities. Proteins are needed as raw materials out of which new cells are made. In addition to these, the body needs water, minerals, and vitamins.

In this experiment a variety of foods will be tested for the presence of water, minerals, starch, sugar, fats, and proteins. For best results in the following tests the food samples should either be ground, or be tested in the shape of scrapings, shavings, or thin slices.

MATERIALS

Test tubes; crucible; beakers (250 ml); watch glass; test tube rack; test-tube holder; filter paper; stirring rod; burner; graduated cylinder; corks or rubber stoppers; concentrated nitric acid (HNO_3); concentrated ammonium hydroxide (NH_4OH); iodine solution; Fehling's solutions (A & B); ether; potato; apple; bread; egg (cooked); banana; raisins; cheese; peanut butter; cobalt chloride paper.

INSTRUCTIONS

Place about 2 ml of freshly scraped potato without peeling into a test tube containing 10 ml of water. Gently boil the mixture for at least one minute, then cool the tube. Try to pour off the liquid so that the sediment remains behind, or if necessary, filter the liquid. Add 1 drop of iodine solution to the liquid. A blue color serves as a positive test for *starch*.

Place about 2 ml of freshly scraped apple into a test tube containing 5 ml of Fehling's solution (equal parts of solutions "A" and "B"). Heat the tube and let the mixture boil gently for a few moments. A red precipitate serves as a positive test for simple *sugars* (monosaccharides).

Place about 2 ml of crumbs from bread into a test tube and add 2 ml of ether. Shake the tube vigorougly for a few minutes. When the solid particles have settled, carefully pour the clear liquid into a very *clean* watch glass. Place the watch glass on the window sill or under the hood while the ether evaporates. The greasy residue which remains on the watch glass is the *fats* which were dissolved from the bread. Place this residue compactly on a piece of paper. Warm the paper for a few moments until the grease melts. Now remove the residue and hold the paper up to the light. This translucent spot is another test for fats. Some foods like peanut butter can be tested in this manner. A translucent spot will appear when the food is scraped off, only if a high percentage of fats are present.

Use a clean, dry stirring rod to place a drop of concentrated nitric acid (HNO_3) on a small piece of cooked (hard boiled) egg white. This color with nitric acid serves as a positive test for *protein*. Wash the food. Now place a drop of ammonium hydroxide on the spot where the acid was previously applied. A deepening color is a further test for the presence of protein.

Place a sample of food in a large tube, stopper loosely, and place in a beaker of boiling water. After about ten minutes remove the tube and examine the interior for condensed water vapor. Test any vapor with cobalt chloride paper. If the blue paper turns pink, *water* is definitely present in cheese.

Place the food tested above in a crucible and heat until all the carbon is burned away. If a grayish-white deposit remains in the crucible, *minerals*, such as iron, calcium, sodium, potassium, phosphates, etc. are present in the cheese. This is a positive test for minerals.

Test all of the food items on the data sheet for the presence of starch, sugar, fat, protein, water, and minerals. Record all observations.

DATA

Food	Starch	Sugar	Protein	Water	Minerals	Fat
Potato						
Apple						
Bread						
Egg White (cooked)						
Banana						
Raisins						
Cheese						
Peanut Butter						
Egg Yolk (cooked)						

EXPERIMENT E-3: ORGANIC MACROMOLECULES--PROTEINS

INTRODUCTION

Proteins are macromolecules ranging in molecular weight from 6,000 to several million. When boiled with mineral acids they yield amino acids. To date 20 amino acids have been isolated from proteins. In spite of the very great diversity of type, there are certain properties common to proteins in general. They are mostly amorphous, although a few crystallize readily and others can be made to do so with difficulty. Some dissolve in water; other, insoluble in water, dissolve in dilute salt solutions. In either case they form colloidal solutions, a fact that is extremely important in the prepara-

tion and maintenance of colloidal states in food preparation. The colloidal nature of proteins is also of great importance in connection with the regulation of cell activities, since this property prevents protein substances from diffusing through membranes, through which some ions pass with ease.

MATERIALS

Cheesecloth; funnel; filter paper; evaporating dish; test tubes; graduated cylinders; eye droppers; 250 ml beaker; 6 M nitric acid (HNO_3); 3 M ammonium hydroxide (NH_4OH); 1% copper sulfate ($CuSO_4$) solution; 3 M sodium hydroxide (NaOH); egg albumin; casein; gelatin; white woolen yarn; white cotton; rolled oats; milk; flour; sausage; 3 M acetic acid ($HC_2H_3O_2$); distilled water.

INSTRUCTIONS

Place a small piece of heat-coagulated egg white in a test tube and add 3 ml of nitric acid (HNO_3). Heat gently until there is a decided color change. Record your observations. Pour off the acid, rinse with distilled water and add 2 ml of ammonium hydroxide (NH_4OH) solution. Observe the deepened color of the egg white due to the formation of the ammonium salt of xanthoproteic acid. Record your observations. Repeat this procedure using woolen yarn, cotton, rolled oats, and sausage. Record your results in the data chart as you perform each test.

The following test is called the Biuret Test. Shake a very small amount of powdered albumin with water. Add an equal volume of 3 M sodium hydroxide (NaOH) solution and one drop of very dilute copper sulfate ($CuSO_4$) solution. Observe the color. Record your observation in the data table. Repeat using a small piece of gelatin which has been soaked in water. Also repeat using casein, a flake of rolled oats, and a small piece of sausage which has been boiled in a small amount of water. In each of the cases the food should be placed in water, then sodium hydroxide added, followed by one drop of the copper sulfate solution. Record all observations on the data table.

Add two milliliters of 3 M acetic acid to about five milliliters of milk. Heat to complete the curdling. Filter and save both the filtrate and the residue. Divide the filtrate into two portions. Test one part with the nitric acid and ammonium hydroxide and compare with the original filtrate. To the second portion of the filtrate add an equal portion of sodium hydroxide and one drop of dilute copper sulfate solution. Record the color changes. Divide the residue into two portions. Run the nitric acid and ammonium hydroxide test (Xanthoproteic Test) on one portion, and Biuret Test on the second portion. Record your results.

Add to a large spoonful of flour in an evaporating dish enough water to make the flour into a stiff dough. Shape the dough into a little ball and let it stand in a beaker of cold water for ten minutes. At the end of this time put the dough in a piece of cheesecloth and wash it under a tap of cold water. Knead thoroughly during the washing process. When the wash water no longer looks milky, open the cheesecloth and examine the product. Apply the Xanthoproteic Test and the Biuret Test to small portions. Record your results in the data table.

If you have brought in any other types of foods from home to test them, apply both the Xanthoproteic Test and the Biuret Test to the substances. Record all of your other observations in the data table. Expand the data table if necessary to include all the observations on all of the substances you have brought in.

DATA

Substance Tested	Xanthoproteic Test After Adding HNO	Acid Test After Adding NH_4OH	Biuret Test
Egg Albumin			
Casein			
Woolen Yarn			
Cotton			
Sausage			
Gelatin			
Oats			
Milk Filtrate			
Milk Residue			
Flour			
Other:_____			

EXPERIMENT E-4: A STUDY OF CARBOHYDRATES AND THEIR REACTIONS

INTRODUCTION

Sugars and starches are the principal sources of energy in the ordinary human diet but they are not essential to the body or to its individual cells in any way, as far as we know. The cells could obtain their energy just as well from a mixture of protein or fat, or even from other types of carbon-containing molecules. Carbohydrates are the cheapest food commercially and this is undoubtedly respon-

sible for the large amounts contained in the average diet. Carbohydrates, although used primarily as an energy source, also supply carbon skeletons (carbon atoms linked together and associated with hydrogen and oxygen) that are necessary in the manufacture of the basic components of protoplasm.

The basic units in carbohydrates are composed of carbon, hydrogen and oxygen. Most, though not all carbohydrates, occur in plants, which have the power of building up these rather complicated molecules from CO_2 and H_2O. In so doing, it is known that they make use of the energy of the sun's rays and that chlorophyll assists in this transformation.

MATERIALS

Sucrose, dextrose, dextrin, starch, maltose and lactose solids; solutions of these carbohydrates; dilute iodine solution; Fehling's solution A and B; concentrated sulfuric acid (H_2SO_4); concentrated hydrochloric acid (HCl); citric acid ($H_3C_6H_5O_7$) solution; 95% alcohol solution; 3 M sodium hydroxide (NaOH) solution; miscellaneous items from home for carbohydrate testing; watch glasses; funnels; filter paper; beakers; glass plate; graduated cylinder; eyedropper; test tubes; corks; burner; litmus paper; wood splints.

INSTRUCTIONS

Place enough starch in a test tube to fill the curved bottom of it. Holding the tube horizontally, heat the starch strongly. Record on the data sheet what you see happening.

Place a drop of concentrated sulfuric acid (H_2SO_4) on a splinter of wood which is mainly cellulose, $C_6H_{10}O_5$. Then add about 1 ml of the concentrated acid to a little sugar in a test tube. Sucrose is $C_{12}H_{22}O_{11}$. These substances are organic compounds which contain the elements of water in combination with carbon. Record all observations.

Place a drop of each of the carbohydrate solutions on a glass plate which has a piece of white paper under it. Add a drop of iodine solution to each drop. Record in the data table what happens in each case. Be careful to label each so that you will not confuse one with the other. Record all observations.

Mix equal volumes (15 ml of each) of Fehling's solutions A and B. Place about 5 ml of this solution in a test tube and heat about to boiling. The solution should remain clear and dark blue. If there are marked color changes, discard this solution, clean out your test tubes thoroughly and try mixing equal volumes again. In each of five test tubes place about 5 ml of solutions of dextrose, sucrose, maltose, dextrin, starch, and lactose. Add about 3 ml of mixed

Fehling's solution to each tube. Place the tubes in a beaker of boiling water and heat for two minutes. Record any color changes in the data table.

Place small quantities of the dry carbohydrates in *dry* test tubes. Add about 5 ml of 95% ethyl alcohol and shake the tubes. Filter the contents through small funnels onto watch glasses and evaporate over a water bath. Any residue left on the watch glass indicates that the carbohydrate is soluble in alcohol. Repeat this procedure using water as a solvent instead of the alcohol solution. Record all of your observations.

In each of three test tubes put 5 ml of sucrose solution. In one add 2 ml of hydrochloric acid (HCl), in a second add 2 ml of citric acid solution, and in the third add 2 ml of water. Place these tubes in a beaker of boiling water and boil for two minutes. Remove the test tubes from the beaker and just neutralize the acid in the first two test tubes with 3 M sodium hydroxide (NaOH), using litmus paper as an indicator. To each tube add some mixed Fehling's solution and return the tubes to the beaker of hot water and heat for three minutes. What observations can you make and record? In this reaction the H^+ from the acids acts as a catalyst.

Optional: Test orange juice, corn syrup, and other items like these with Fehling's mixed solution and technique used above to determine which of these substances contain glucose sugar.

DATA

Observations	
Starch + Heat	
Cellulose + Sulfuric Acid (conc.)	
Sucrose + Sulfuric Acid (conc.)	

Carbohydrate	Iodine Test	Fehling Test	Solubility 95% Alcohol	Solubility Water
Sucrose				
Dextrose				
Dextrin				
Maltose				
Lactose				
Starch				

Optional Results	
Substance Tested	Presence of Glucose
1.	
2.	
3.	
4.	
5.	

EXPERIMENT E-5: THE DIGESTION OF STARCH

INTRODUCTION

In this experiment you will be studying a specific carbohydrate, starch, which is a polysaccharide. Starch has the formula $(C_6H_{10}O_5)_x$ where x is a very large number. This polysaccharide which we eat must be broken up into simpler substances before it can be assimilated and used. We will be investigating the effect of two substances in the digestive system on starch--saliva and pancreatic extract.

MATERIALS

Soluble starch; pancreatic extract solution; iodine solution; Fehling's solution A and B; 5% iron (III) chloride ($FeCl_3$) solution; mercury (II) chloride ($HgCl_2$) solution; distilled water; hydrochloric acid (HCl); paraffin; pH paper; glass plates; wax pencils; evaporating dishes; eyedropper; thermometer (°C); white paper; graduated cylinder; burner; assorted beakers; ring and ringstand; wire gauze; timer; assorted test tubes.

INSTRUCTIONS

Prepare your starch solution by adding 1.0 grams of soluble starch to 10 ml of water, stirring, and adding to 100 ml of boiling water. Allow this starch solution to cool to 30°C. Next prepare a Fehling solution mixture by mixing 3 ml of Fehling's solution A with 3 ml of Fehling's solution B and 6 ml of water. Mix thoroughly.

Place a glass plate over a sheet of white paper and mark off twelve squares on it with a marking pencil. Then place twelve separate drops of iodine solution on it, one in each square. Collect some of your saliva in a beaker. If you have difficulty producing enough saliva, chew on a piece of paraffin.

In order to have a control in this experiment, test one drop of the *cooled* starch paste with one drop of the iodine solution. Also test a small portion of the starch solution with some Fehling's mixture to determine the presence or absence of reducing sugar at the beginning of the experiment. (If no sugar is present, the solution will remain clear blue. If a trace of sugar is present the solution will become green. As the amount of sugar increases, the color should change until it finally becomes red.) Save these control tests in order to compare them with the "digested" tubes later in the experiment.

Fill a large test tube one-third full of *cooled* starch solution. Add approximately 3 ml of saliva. Mix. At the end of three minutes, place one drop of the starch-saliva mixture on one drop of the iodine solution on the glass plate. Record the color change. Add about 2 ml of this starch-saliva mixture to 2 ml of the Fehling's solution mixture and heat in the hot water bath. If the starch has been hydrolyzed by the enzyme– amylase – in saliva, a color change should be noticeable.

Repeat the iodine test and the Fehling's solution test at intervals of three minutes until the iodine test is negative and the Fehling's test is positive. Record all of your observations in the data chart.

Fill a second clean test tube one-third full of *cooled* starch solution. Add about 5 ml of pancreatic extract solution. Mix. At the end of three minutes run the iodine solution and Fehling's solution tests as you did on the starch-saliva mixture earlier. Record all results in the data chart. Repeat the tests at three minute intervals until the iodine test is negative and the Fehling's test is positive, as before.

Place a drop of saliva on pH paper and estimate the pH value of it. Do the same for the pancreatic extract solution. Record this data.

Optional Tests on Saliva

An approximation of the amount of solid enzyme amylase present in saliva may be made by a simple evaporation. Place 3 ml of saliva on a microscope slide or a glass plate. Encircle the 3 ml with a ring made with a wax marking pencil to prevent the contents from flowing off. Warm to dryness over a beaker of boiling water. Observe and note the amount of residue.

Place 5 ml of saliva into each of two evaporating dishes. In a third dish put 5 ml of water to serve as a blank. To each dish add a few drops of a 5% iron (III) chloride ($FeCl_3$) solution and acidify each slightly with hydrochloric acid (HCl). Add 4 drops of mercury (II) chloride ($HgCl_2$) solution to one of the evaporating dishes that contains saliva. Stir. Notice the final coloration. *Note:* Some foods

in your diet, notably cherries, increase the excretion of thiocyanates in the saliva. Smoking increases the thiocyanate in the saliva. In fact there is a definite relationship between the deepness of the red color from the thiocyanate ion reaction with the iron III ion and the number of cigarettes smoked per day.

DATA

	Saliva		Pancreatic Extract	
Time	Iodine Test	Fehling 's Test	Iodine Test	Fehling's Test
Control				
3 min.				
6 min.				
9 min.				
12 min.				
15 min.				
18 min.				
etc.				

	pH
saliva	
pancreatic extract	

EXPERIMENT E-6: CONSUMER FOOD TEST I
BUTTER AND MARGARINE

INTRODUCTION

Butter has been used for various purposes since 2000 BC or before. Its use as a food is modern. In the early centuries butter was offered as a sacrifice in worship or used for medical purposes in skin diseases. In the United States, butter has always been used as a food. Most of the butter on the market is made from pasteurized cream to which a starter culture of bacteria has been added. Salt is added because it helps in the removal of buttermilk, enhances the flavor of butter, and improves its keeping qualities. The Federal

Food, Drug, and Cosmetic Act requires butter to contain not less than 80% by weight of milk fat and to be made from clean, unspoiled cream. In this experiment we will test butter for the presence of artificial colors, spoilage indicators, and attempt to observe some differences between butter and margarine.

MATERIALS

Butter; margarine; ice; ether; phenolphthalein; 0.05 M sodium ethylate solution; carbon tetrachloride (CCl_4); ethyl alcohol (C_2H_5OH); concentrated ammonium hydroxide (NH_4OH); concentrated sulfuric acid (H_2SO_4); 125 ml Erlenmeyer flask; centigrade thermometer; screen; assorted beakers; timer; separatory funnel; buret; buret clamp; ring and ringstand; burner; test tubes; graduated cylinder.

INSTRUCTIONS

To distinguish between pure butter and renovated butter or margarine, melt one teaspoon of butter and one teaspoon of margarine in separate test tubes. Observe how each melts, whether or not foam is produced, and record your observations.

To detect whether there is artificial coloring present in butter and margarine, place a half teaspoon of butter in a flask. Add 5 ml of carbon tetrachloride (CCl_4) and 10 ml of ethyl alcohol (C_2H_5OH). Shake for a minute. Let the mixture stand until it separates. The lower carbon tetrachloride layer contains the fat and the upper alcohol layer any artificial coloring matter. If the alcohol layer is colorless, there is no artificial coloring matter in the butter or margarine. If the alcohol layer is colored, pour 5 ml of it into a test tube and add 10 drops of ammonium hydroxide (NH_4OH), (concentrated). A brown color indicates that turmeric is the coloring matter. Evaporate the remainder of the alcohol layer to dryness and add a drop of concentrated sulfuric acid (H_2SO_4) to the residue. A greenish-blue color indicates the presence of annatto as a coloring agent. Repeat these tests for butter and margarine. Record all of your observations on the data sheet.

To determine whether butter was made from decomposed cream, weigh out 10 grams of the butter and place it in a 125 ml Erlenmeyer flask. Heat the flask gently until the butter melts, then cool until the butter is thick and creamy. Add 50 ml of ice water to the butter in the flask and shake for five seconds. Immerse the flask in an ice bath until the temperature is 10°C. Remove the thermometer and shake the flask for another five seconds.

Place a screen over the neck of the flask and pour off and discard the liquid layer through the screen. Add 50 ml of ice water through the screen to the butter in the flask and again shake for five seconds. Pour off the water layer again. Repeat this procedure with

three more portions of ice water, each 50 ml in volume. After the fifth washing add 25 ml of ether to the butter and swirl the flask until the butter dissolves. Transfer the solution to a separatory funnel. Wash the flask with 2-3 ml more of ether and add the washings to the separatory funnel. Let the funnel stand for a few minutes. Then drain off the lower aqueous layer and discard it. Place the remaining ether-fat solution from the funnel to a 125 ml Erlenmeyer flask. Wash the funnel with a few mls of ether and transfer the washings to the flask. Titrate this ether-fat solution with 0.05 M sodium ethylate solution until the first permanent pink color appears. Record the volume of sodium ethylate solution used.

DATA

Melting Observations:	
Butter	Margarine
Brand:_____	Brand:_____

Test for Artificial Coloring Additives				
	Brand Name	Present	Not Present	Color
Butter				
Margarine				

Titration of Butter vs. Sodium Ethylate	
Final Buret Reading	_____ ml
Initial Buret Reading	_____ ml
Volume of Sodium Ethylate Used	_____ ml

Titration Calculations:

1 ml of 0.05 M sodium ethylate solution is equivalent to 13.5 mg of water-insoluble acids in butter.

Volume of sodium ethylate used × 13.5 mg = no. of mg of water insoluble acids in 10 grams of butter

_____ml × 13.5 mg = _____mg

_____mg × 10 = _____(no. of mg of water-insoluble acids in 100 grams of butter)

Butter made from acceptable cream will not contain more than 400 mg of water-insoluble acids per 100 grams of butter. Was the butter you tested made from cream acceptable under the requirements?

EXPERIMENT E-7: CONSUMER FOOD TESTS II
CAFFEINE

INTRODUCTION

Coffee is one of the most popular beverages of the world. The quality of a cup of coffee depends partly upon the kind of coffee and partly upon how it is made. A cup of coffee will contain the water-soluble products of the berry, the most important of these being tannin, caffeine, caffeol, caramelized carbohydrates, preservatives, cereals, and carbon dioxide. Tannin dissolves slowly in hot water. The bitterness of some coffee is due to the extraction of too much tannin. Caffeine is the alkaloid which may act as a stimulant. Every cup of coffee contains caffeine unless this has been removed from the berry before brewing. Carmelized carbohydrates and other decomposition products formed in the roasting process are brown in color and account for the color of the brew.

Tea is a water solution of the soluble products in the tea leaf. Of these the most important are caffeine, tannin, soluble colored substances, and the essential oils which impart the aroma and flavor. The amount of any of these products will depend upon the method of brewing. The percentage of caffeine in tea is actually greater than in coffee, but the amount present in a cup of tea is usually less, as most people prefer a very weak brew.

MATERIALS

Various brands of coffee, tea, soft drinks, and "stay awake" tablets; chloroform; ethyl alcohol; distilled water; iodine solution; concentrated ammonium hydroxide (NH_4OH); graduated cylinder; separatory funnels; watch glass; beakers; timer; burner; ring and ringstand; funnel; filter paper; mortar and pestles; melting point apparatus.

INSTRUCTIONS

Place one teaspoon of coffee carefully on the top of a beaker of *cold* water. Real coffee will float and will not discolor the water for a few minutes. Most adulterants used in coffee sink quickly to the bottom, discoloring the water. Starchy adulterants such as roasted cereals can be detected. If the solution becomes discolored immediately after adding the coffee, boil the coffee and the water. Stir the mixture for a few minutes. Then filter the solution. Dilute the coffee solution until it is a light amber color and add iodine solution. A blue color indicates starchy cereals have been added to that brand of coffee. If the coffee floats and the solution does not discolor, proceed with the test for caffeine.

Boil the coffee-water mixture for 15 minutes. Cool and filter if necessary. Add 30 ml of chloroform and transfer the entire mixture to a separatory funnel. Stopper and shake well. Allow the layers to separate. Remove the bottom chloroform layer into a beaker. Let the liquid evaporate in a water bath. Then dissolve the residue in 10 ml of ethyl alcohol. Evaporate to dryness on a watch glass over a water bath. Observe the residue for caffeine crystals—they should be white long needles in their crystal form. *Optional:* Using the procedure learned in two previous experiments B-6 and C-3, place some of the caffeine crystals in a capillary tube and determine its melting point.

The extraction procedure with chloroform and ethyl alcohol should now be repeated with other brands of coffee, tea, soft drinks, colas, and "stay awake" tablets. In the case of the extraction from cola beverages, 10 ml of concentrated ammonium hydroxide (NH_4OH) should be added and mixed well for several minutes before adding the chloroform. When the extraction of caffeine is done on the tablets, one tablet should be crushed in a clean mortar and pestle. Then the powder should be stirred into 50 ml of distilled water. If the solution is cloudy, this is caused by the undissolved filler in the tablet and need not be filtered. Observations on the amount of caffein present in these household commodities should be recorded in the data sheet.

DATA

Item	Brand Name	Presence of Starch	Presence of Caffeine
1.			
2.			
3.			
4.			
etc.			

Sketch of Caffeine Crystals:
Optional: Melting Point of Caffeine Crystals = _____ °C

EXPERIMENT E-8: CONSUMER FOOD TESTS III
SYNTHETIC DYES

INTRODUCTION

To make foods more appealing, artificial colors are often added. However, only colors approved by the Food and Drug Administration (FDA) are permitted, and the requirements are: that the food must not appear better than it actually is, the color must not be toxic, and the presence of the color must be stated on the label. In this experiment, various foods will be tested for the presence of coal-tar and synthetic dyes. The techniques will be qualitative and quantitative. In the first section the presence of a dye will be checked out. Then, using the technique of paper chromatography, the dye will be analyzed by comparing it to standard certified FDA colors.

MATERIALS

Various consumer products (catsup, jam, jelly, pop, gelatin desert, candy, gum, oranges, etc.); white wool yarn pieces; normal butyl alcohol (C_4H_9OH); hexane (C_6H_{14}); saturated ammonium sulfate ($(NH_4)_2SO_4$) solution; blue litmus paper; 1% acetic acid; 10% LA-2 amberlite resin solution in butyl alcohol; 1% sodium chloride (NaCl) solution; 0.1% solutions of FDA certified colors; 10% ammonium hydroxide solution (NH_4OH); concentrated hydrochloric acid (HCl); dilute hydrochloric acid (1:2); cheesecloth; separatory funnel; small beakers; capillary tubes; test tubes; paper clips; corks; graduated cylinder; ring and ringstand.

INSTRUCTIONS

Use about a tablespoonful of the food to be tested (catsup, jam, or jelly) and mix thoroughly with about 150 ml of water. Filter through cheesecloth. Add three drops of concentrated hydrochloric acid and heat gently in a beaker. Place a piece of white woolen yarn into

the mixture and heat for about 10 minutes. Remove the yarn and rinse in clear water. If the yarn has become colored, dyes are present in the food. To confirm this test, dissolve the aniline-based color out of the yarn with about 100 ml of a dilute solution of ammonium hydroxide (10%). Natural fruit color on the yarn will not be affected. Neutralize ammonium hydroxide (NH_4OH) solution with dilute hydrocloric acid (use blue litmus to test that it is slightly acidic). Then stir a fresh piece of yarn in this acid solution. If this second piece of yarn is dyed and retains its color when rinsed in water, aniline dyes are present in the food tested. Repeat the test on other foods and record your results.

In order to separate and identify the dyes present in foods, the following technique of chromatography will be practiced and then applied to the analysis of dyes found in various foods.

In order to run chromatograms of the standard colors certified by the Federal Food and Drug Administration for addition to foods, prepare four test tubes as is shown in Figure E-8. Chromatographic paper or filter paper can be cut into the proper width and lengths for the strips to be hung in the test tubes. Use an open capillary tube for spotting the papers. Spot the first four papers with FD&C Blue No. 1, FD&C Green No. 3, FD&C Yellow No. 5, and FD&C Yellow No. 6, about 1 inch from the edge of the strip. Allow the spots to dry. Add 1% sodium chloride (NaCl) solution to the tubes to a depth of 1/4 to 1/2 inch. Suspend the paper in the tube so that the lower edge touches the liquid, but so that the liquid is below the spots. Cork the tube and allow the chromatograms to develop until the solvent has moved approximately to the top of the paper. Remove and dry, not forgetting to label the papers after they dry. Place spots of FD&C Violet No. 1, FD&C Red No. 2, FD&C Red No. 3, and FD&C Red No. 4 on four more pieces of paper and place them in the tubes allowing the solvent to move up to the same location before removing them for drying. Remember to label them. After all eight strips are dry and labeled, lay them edge to edge and tape the backsides together so that you can compare all the standards in one glance. Set these standards aside until you are ready to compare the unknown dyes in the foods which you are now ready to test.

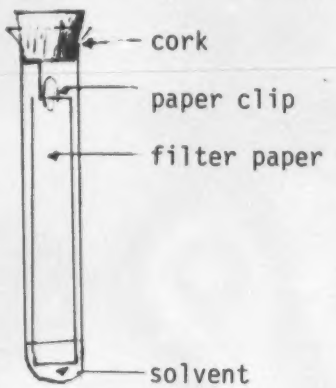


Figure E-8.

Select an artificially-colored beverage, such as grape soda. Pour approximately 25 ml into the separatory funnel. Add 10 ml of the resin solution. Shake the separatory funnel gently until the color transfers to the upper (resin in butyl alcohol) layer. A 1/2 ml of saturated ammonium sulfate ($(NH_4)_2SO_4$) solution should separate any emulsion formed by a too vigorous shaking. Drain off and discard the lower layer. Place the resin extract from the beverage into a corked test tube.

Dissolve 2 grams of a strongly-colored gelatin dessert in 20 ml of hot water. After cooling, transfer the solution to a separatory funnel and extract the color in the same manner as you did with the beverage, storing it in a corked test tube.

Dissolve 2 grams of a strongly-colored hard candy in 20 ml of 1% acetic acid ($HC_2H_3O_2$) and extract the color in the same manner as above.

Shake each of the resin layers extracted above with one to three 10 ml portions of water until the lower layer is not acidic to blue litmus paper. Discard the washes. Mix 20 ml of hexane (C_6H_{14}) with each of the resin layers and discard any water layer that separates.

Extract the color from the hexane-butyl alcohol resin layer by shaking gently with 2 ml portions of 10% ammonium hydroxide (NH_4OH). No more than three shakings should be required to remove all of the color. Collect the color solution in a small beaker. Spot the unknown dyes extracted from the foods about 1 inch from the edge of a strip if paper. These dilute solutions will make a more concentrated spot by multiple spotting. To keep the spot small allow each spotting to dry before adding the next drop of color to the same area.

Chromatograph the mixtures of unknown colors in the same way that you chromatographed the FD&C standard food colors. After the strips are dry and labeled compare them to the standards and record all of your results.

Optional: If you wish to test the color added to oranges, the following procedure is given. Place a flask beneath a funnel and support an orange on some glass rods. Wash the color off the orange by spraying it with 25 ml of chloroform in a fine stream--use a pipet with a bulb. Surface oils, waxes and natural pigments as well as the artificial color will be washed off. Repeat the step above with two or more of the oranges and combine the washings in the same flask.

Transfer the solution to a small beaker. Allow the solution to evaporate by placing the beaker in a steam-hot water bath. Allow the beaker to cool after removing it from the steam bath. Dissolve the residue in 3 ml of chloroform. Spot a sheet of chromatography paper with this color extract. If approved Citrus Red No. 2 and Oil Red XO FD&C colors are available, spot papers with solutions of these. Place all spotted papers in the test tubes. After the solvent has traveled on the papers to the top, remove them, dry them, and label them for comparisons.

DATA

Observations of Wool Yarn Test	Food #1	Food #2	Food #3	Food #4	Food #5
Color of White Yarn Piece:					
Type of Food Tested:					
Was Aniline Dye Present?					

Chromatography Results								
Type of Foods Tested	FD&C Certified Food Colors Present							
	Blue #1	Green #3	Violet #1	Red #2	Red #3	Red #4	Yellow #5	Yellow #6
1.								
2.								
3.								
4.								
5.								

EXPERIMENT E-9: DETERMINATION OF CITRIC ACID AND VITAMIN C IN FRUIT JUICES

INTRODUCTION

Oranges and other citrus fruits should not contain more than 5% of citric acid ($H_3C_6H_5O_7$). The percentage of citric acid in juices may be determined by neutralizing it with standard NaOH, using phenolphthalein as an indicator. Vitamin C, or ascorbic acid ($C_6H_8O_6$), is a sugar-like compound that is readily oxidized. In this experiment you will oxidize it by titrating a known volume of juice with

very dilute iodine solution, using starch as an indicator. The iodine will not react with the starch until all the ascorbic acid has been oxidized.

MATERIALS

Phenolphthalein, 0.20 M sodium hydroxide (NaOH); fresh vitamin C tablets; iodine solution; starch indicator; samples of fruit juices; dilute hydrochloric acid (1:4); distilled water; pipet; buret clamp; ringstand; graduated cylinder; misc. beakers; 250 ml Erlenmeyer flask; mortar and pestle; eye dropper; timer.

INSTRUCTIONS

Set up a clean buret and fill it with 0.20 M sodium hydroxide (NaOH). Then pipet a sample of the orange juice being tested into a clean Erlenmeyer flask. Add an equal amount of distilled water and three drops of phenolphthalein. Place a sheet of white paper underneath the flask. Titrate the citric acid with the sodium hydroxide solution until an endpoint is reached, which is a pink color lasting for 30 seconds. Record your data. Repeat this procedure with two other samples of orange juice.

Pulverize a tablet of vitamin C in a mortar. Place the powder in a flask and wash it with 5 ml of distilled water. Make sure you transfer all of the tablet. Acidify the material in the flask with two drops of dilute (1:4) hydrochloric acid. Add 3 ml of starch solution indicator in the flask. Titrate this solution with a standard stock iodine solution using the buret until a faint blue-violet color persists with swirling. Make a careful record of your buret readings. Repeat this titration with two other brands. Don't forget to record on your data sheet the information from the label stating the number of mg of vitamin C present in each of the tablets.

Repeat the above vitamin C titration with a sample of an "Unknown" --orange juice, grapefruit juice, lemon juice, etc. Vitamin C is unstable and easily destroyed by heating or leaving exposed to air. Therefore, titrate another sample of juice after heating it in a beaker before titration.

DATA

Citric Acid Titration			
	Sample #1	Sample #2	Sample #3
Final Buret Reading			
Initial Buret Reading			
Volume of 0.20 M NaOH Used			

Vitamin C Tablet Analysis			
	Brand #1	Brand #2	Brand #3
Number of mg Present on Label			
Final Buret Reading			
Initial Buret Reading			
Volume of Standard Iodine Solution Used			

Juice Analysis for Vitamin C		
	Unheated Sample	Heated Sample
Final Buret Reading		
Initial Buret Reading		
Volume of Standard Iodine Solution Used		

EXPERIMENT E-10: A STUDY OF THE pH OF COMMON FOODS

The acidity of a solution is a vital factor which determines the outcome of many reactions. People who have swimming pools carefully control the acidity so as to avoid undesirable reactions with the plumbing and plaster and to expedite reactions with other chemicals. The food, drug, petroleum, and most chemical industries continually monitor the acidity of the solutions being used in the preparation of their products.

The pH scale is a numerical scale which, for most applications, extends from 0 through 14. The numbers on the scale represent the *relative* acidity of solutions. The midpoint of the scale is taken as 7.0. A solution with a pH of 7.0 contains equal concentrations of H^+ and OH^- ions and is neutral. A solution with a pH less than 7.0 has a greater H^+ than OH^- ion concentration and is acidic. Solutions with pH's above 7.0 have greater OH^- than H^+ ion concentrations and are basic. In this experiment, you will prepare your own indicator and test various foods to determine the relative acidity of each.

MATERIALS

Red cabbage leaves; distilled water; buffer solutions of various pH's; vinegar; salt water; lemon juice; apple juice; milk; orange juice; ginger ale; sauerkraut juice; coffee; tea; sugar; red and blue litmus paper; vanilla flavoring; flour; baking soda; grapefruit juice; baking powder; colored pencils or crayons; assorted beakers; small test tubes; burner; ring and ringstand; graduated cylinder.

INSTRUCTIONS

Shred a leaf of red-purple cabbage into a small beaker and cover with distilled water. Boil the cabbage in the water until the solution is a deep purple. Obtain 3 ml of all of the pH buffer solutions and place in test tubes. Then add enough of the cabbage-juice indicator until you can tell its color. Record this color on the data sheet and fill in the color chart with crayons or colored pencils. Save these tubes since they will be your standards for comparisons with the solutions of various foods.

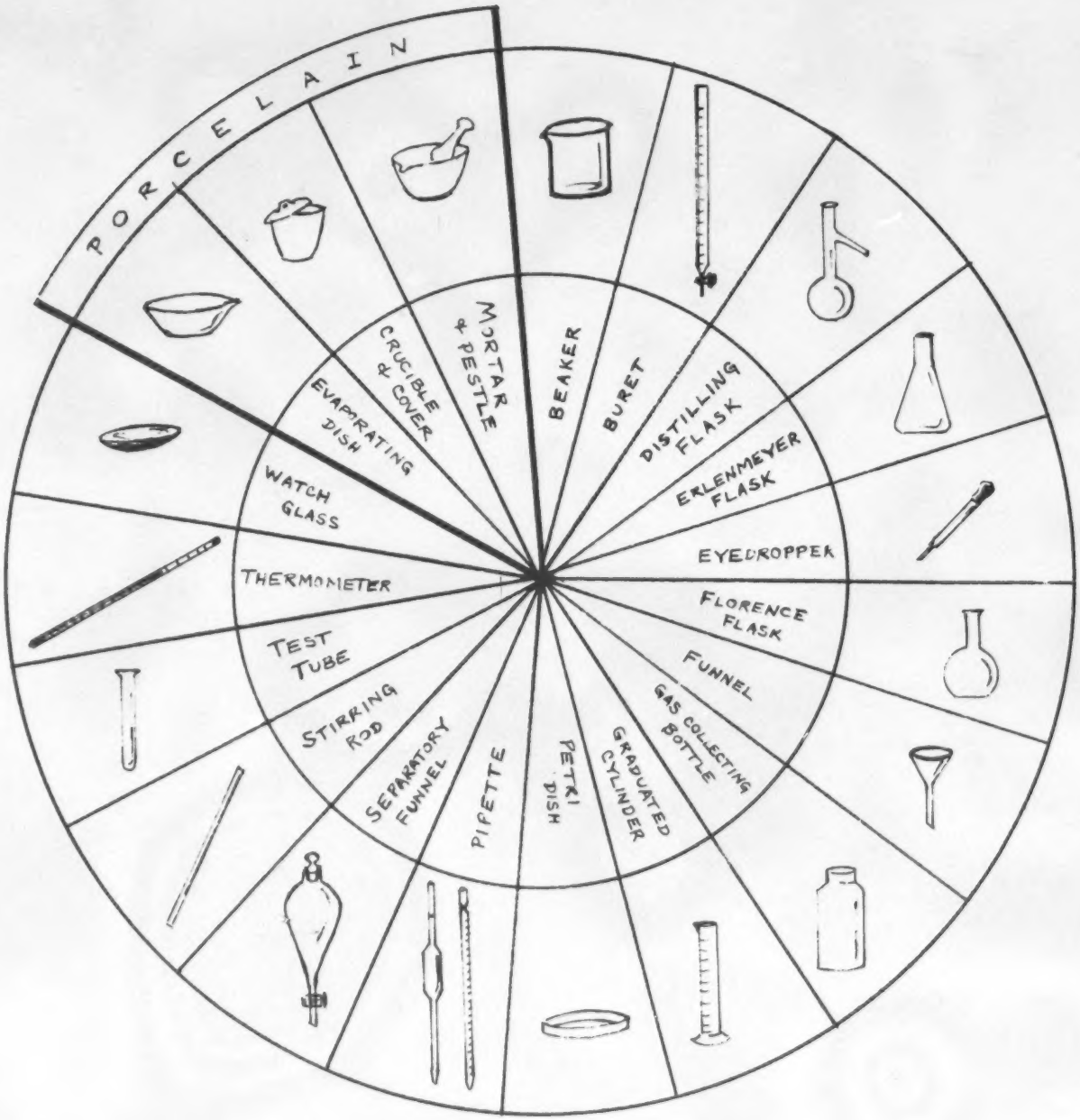
Obtain 3 ml of the various solutions listed in the second data table and place in your test tubes. First test the solution with red and blue litmus paper to determine whether it is acidic or basic. Record this information on the data table. Then add some of the cabbage-juice indicator to the solutions and determine the pH of these solutions by comparisons with the standards prepared earlier. If the original food solution you are testing is already colored, take this into account when determining its pH. Record all data.

DATA

Colorimetric pH Changes for Cabbage-Juice Indicator Standards

pH of Buffer Solution	Acidic	Basic	Neutral	Color Description	Color Chart
0					
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					

pH of Common Foods						
	Acidic	Basic	Neutral	Original Color	Color With Indicator	pH
Vinegar						
Salt Water						
Lemon Juice						
Apple Juice						
Milk						
Orange Juice						
Ginger Ale						
Sauerkraut						
Baking Powder						
Baking Soda						
Coffee						
Tea						
Vanilla Flavor.						
Saliva						
Sugar Water						
Distilled Water						
Tap Water						
Flour						
Grapefruit Juice						



LABORATORY EQUIPMENT: GLASS AND PORCELAIN

LABORATORY EQUIPMENT: METAL, PLASTIC AND RUBBER

LIST OF ELEMENTS, NAMES, ATOMIC NUMBERS, AND ATOMIC WEIGHTS

NAME	SYMBOL	ATOMIC NUMBER	ATOMIC WEIGHT	NAME	SYMBOL	ATOMIC NUMBER	ATOMIC WEIGHT
Actinium	Ac	89	(227)	Mercury	Hg	80	200.6
Aluminum	Al	13	27.0	Molybdenum	Mo	42	95.9
Americium	Am	95	(243)	Neodymium	Nd	60	144.2
Antimony	Sb	51	121.8	Neon	Ne	10	20.2
Argon	Ar	18	39.9	Neptunium	Np	93	(237)
Arsenic	As	33	74.9	Nickel	Ni	28	58.7
Astatine	At	85	(210)	Niobium	Nb	41	92.9
Barium	Ba	56	137.3	Nitrogen	N	7	14.01
Berkelium	Bk	97	245	Osmium	Os	76	190.2
Beryllium	Be	4	9.01	Oxygen	O	8	16.00
Bismuth	Bi	83	209.0	Palladium	Pd	46	106.4
Boron	B	5	10.8	Phosphorus	P	15	31.0
Bromine	Br	35	79.9	Platinum	Pt	78	195.1
Cadmium	Cd	48	112.4	Plutonium	Pu	94	(242)
Calcium	Ca	20	40.1	Polonium	Po	84	210
Californium	Cf	98	(251)	Potassium	K	19	39.1
Carbon	C	6	12.01	Praseodymium	Pr	59	140.9
Cerium	Ce	58	140.1	Promethium	Pm	61	(147)
Cesium	Cs	55	132.9	Protactinium	Pa	91	(231)
Chlorine	Cl	17	35.5	Radium	Ra	88	(226)
Chromium	Cr	24	52.0	Radon	Rn	86	(222)
Cobalt	Co	27	58.9	Rhenium	Re	75	186.2
Copper	Cu	29	63.5	Rhodium	Rh	45	102.9
Curium	Cm	96	(247)	Rubidium	Rb	37	85.5
Dysprosium	Dy	66	162.5	Ruthenium	Ru	44	101.1
Einsteinium	Es	99	(254)	Samarium	Sm	62	150.4
Erbium	Er	68	167.3	Scandium	Sc	21	45.0
Europium	Ei	63	152.0	Selenium	Se	34	79.0
Fermium	Fm	100	(253)	Silicon	Si	14	28.1
Fluorine	F	9	19.0	Silver	Ag	47	107.9
Francium	Fr	87	(223)	Sodium	Na	11	23.0
Gadolinium	Gd	64	157.3	Strontium	Sr	38	87.6
Gallium	Ga	31	69.7	Sulfur	S	16	32.1
Germanium	Ge	32	72.6	Tantalum	Ta	73	180.9
Gold	Au	79	197.0	Technetium	Tc	43	(99)
Hafnium	Hf	72	178.5	Tellurium	Te	52	127.6
Helium	He	2	4.0	Terbium	Tb	65	158.9
Holmium	Ho	67	164.9	Thallium	Tl	81	204.4
Hydrogen	H	1	1.008	Thorium	Th	90	232.0
Indium	In	49	114.8	Thulium	Tm	69	168.9
Iodine	I	53	126.9	Tin	Sn	50	118.7
Iridium	Ir	77	192.2	Titanium	Ti	22	47.9
Iron	Fe	26	55.8	Tungsten	W	74	183.9
Krypton	Kr	36	83.3	Uranium	U	92	238.0
Lanthanum	La	57	138.9	Vanadium	V	23	50.9
Lead	Pb	82	207.2	Xenon	Xe	54	131.3
Lithium	Li	3	6.94	Ytterbium	Yb	70	173.0
Lutetium	Lu	71	175.0	Yttrium	Y	39	88.9
Magnesium	Mg	12	24.3	Zinc	Zn	30	65.4
Manganese	Mn	25	54.9	Zirconium	Zr	40	91.2
Mendelevium	Md	101	(256)				

Paenthetical atomic weights refer to radioactive elements; the mass number (not the atomic weight) of the isotope with largest half-life is usually given.

NAMES, FORMULAS AND CHARGES OF SOME COMMON IONS

POSITIVE IONS (CATIONS)		NEGATIVE IONS (ANIONS)	
aluminum	Al^{+3}	acetate	$C_2H_3O_2^-$
ammonium	NH_4^+	bromide	Br^-
barium	Ba^{+2}	carbonate	CO_3^{-2}
calcium	Ca^{+2}	hydrogen carbonate ion, bicarbonate	HCO_3^-
chromium (II), chromous	Cr^{+2}	chlorate	ClO_3^-
chromium (III), chromic	Cr^{+3}	chloride	Cl^-
copper (I), cuprous	Cu^+	chromate	CrO_4^{-2}
copper (II), cupric	Cu^{+2}	dichromate	$Cr_2O_7^{-2}$
hydrogen	H^+	fluoride	F^-
iron (II), ferrous	Fe^{+2}	hydroxide	OH^-
iron (III), ferric	Fe^{+3}	iodide	I^-
lead (II)	Pb^{+2}	nitrate	NO_3^-
lithium	Li^+	nitrite	NO_2^-
magnesium	Mg^{+2}	oxalate	$C_2O_4^{-2}$
manganese (II), manganous	Mn^{+2}	permanganate	MnO_4^-
mercury (I), mercurous	Hg^+	phosphate	PO_4^{-3}
mercury (II), mercuric	Hg^{+2}	monohydrogen phosphate	HPO_4^{-2}
potassium	K^+	dihydrogen phosphate	$H_2PO_4^-$
silver	Ag^+	sulfate	SO_4^{-2}
sodium	Na^+	hydrogen sulfate ion, bisulfate	HSO_4^-
tin (II), stannous	Sn^{+2}	sulfide	S^{-2}
tin (IV), stannic	Sn^{+4}	hydrogen sulfide ion, bisulfide	HS^-
zinc	Zn^{+2}	sulfite	SO_3^{-2}
		hydrogen sulfite ion, bisulfite	HSO_3^-